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APPENDICES & ATTACHMENTS

for the

VASQUEZ BOULEVARD & I-70 SITE DENVER, CO

PHASE III FIELD INVESTIGATION

June 30, 1999



Prepared For:
U.S. Environmental Protection Agency, Region 8
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Appendix A:

Selection of Chemicals of Potential Human Health Concern at the Vasquez Boulevard and I-70 Site

SELECTION OF CHEMICALS OF POTENTIAL HUMAN HEALTH CONCERN AT THE VASQUEZ BOULEVARD AND I-70 SITE

1.0 INTRODUCTION

Chemicals of potential concern (COPCs) are chemicals which a) are present at a site, b) occur at concentrations which are or might be of health concern to exposed humans, and c) are or might be due to releases from a Superfund site. USEPA has derived a standard method for selecting COPCs at a site, as detailed in Risk Assessment Guidance for Superfund: Human Health Evaluation Manual (Part A) (USEPA 1989). In brief, USEPA assumes that any chemical detected at a site is a candidate for selection as a COPC, but identifies a number of alternative methods that may be used for determining when a chemical is not of concern and may be eliminated from further consideration. Each risk assessment may choose to apply some or all of the methods identified by USEPA to select COPCs, as appropriate.

2.0 DATA BASE USED TO SELECT COPCs

Most soil samples collected from the Vasquez Boulevard and I-70 (VBI70) site have been analyzed by X-ray fluorescence (XRF) for only a few contaminants (arsenic, lead, cadmium and zinc), and only the data for arsenic and lead are considered to be reliable (UOS 1998). However, a sub-set of samples have been analyzed for the full suite of 23 metals on USEPA's Target Analyte List (TAL), and these data are the basis of the COPC selection procedure. The data consist of two sub-sets:

- Analyses performed during Phase I, using the bulk fraction (particle size <2 mm) of 44 samples selected at random to confirm the accuracy of the XRF measurements for arsenic and lead and to quantify the levels of TAL metals. Because these samples were selected a priori and without regard to the level of contamination, there are only 9 of these samples that contain concentrations of arsenic above 100 ppm, with the maximum value being 1,200 ppm. Thus, these samples are helpful in the COPC selection procedure, but may not necessarily characterize the chemicals that may be of concern at the most contaminated properties.
- Subsequent to Phase I and Phase II, USEPA performed a study on 8 residential properties in the study area specifically intended to support the COPC selection procedure. Five (5) properties were chosen because arsenic levels exceed the established removal action level. Three (3) properties had arsenic below this action level. The data set used to support the COPC selection procedure consists of the fines fraction (particle size <250 μm) of 10 soil samples selected (based on the results of Phase I) to contain high levels of arsenic (6,000 to 12,000 ppm). Two (2) samples from each of the 5 most contaminated properties were chosen because these samples are likely to reflect the contaminants most likely to be of concern.

These data are presented in Appendix 1, and summary statistics are presented in Table 1.

In the case of copper, there is one sample whose analytical value (14,000 ppm) appears to be clearly inconsistent with all of the other 53 values (average = 37 ppm, max = 71 ppm). On this basis, the one extreme value for copper is excluded as an outlier, and screening is based on the remaining samples. All other data values were used. Non-detects were evaluated using the reported detection limit.

3.0 COPC SELECTION METHOD AT THE VBI70 SITE

Step 1: Eliminate chemicals whose maximum value is below a level of concern

This step involves comparing the maximum detected value in a medium to an appropriate Risk-Based Concentration (RBC). If the maximum value is less than the RBC, the chemical does not pose a risk and can be eliminated.

The RBCs used in this evaluation were taken from USEPA's Region III Risk-Based Concentration (RBC) table for residential soil. The value of each RBC depends on the specified Target Risk level. In accord with the goal that the COPC selection process should be conservative, the Target Risk levels used in this evaluation are 1E-06 for carcinogenic chemicals and a hazard quotient (HQ) of 1.0 for noncarcinogenic chemicals.

Table 2 lists the Region III RBCs for each chemical and identifies those which can and cannot be eliminated at this step. Based on this screening step, the following chemicals are eliminated:

- Aluminum
- Barium
- Beryllium
- Cadmium
- Chromium
- Cobalt
- Copper
- Manganese
- Mercury
- Nickel
- Selenium
- Silver
- Vanadium
- Zinc

Step 2. Eliminate Beneficial Minerals

In accord with USEPA (1989), chemicals that are normal constituents of the body and the diet and are required for good health may be eliminated unless there is evidence that site-specific releases have elevated concentrations into a range where intakes would be potentially toxic. At this site, there is no reason to suspect this is the case, so the following chemicals are eliminated on this basis:

- Calcium
- Magnesium
- Potassium
- Sodium

Iron is also eliminated on this basis, since the average concentration of iron (13,400 ppm) is well below the screening level of 23,000 ppm. Additionally, only 1 of 54 samples exceeds the RBC for iron, and this only by a small amount (26,000 vs. 23,000 ppm).

Step 3. Eliminate Chemicals Whose Contribution is Minor Compared to Others

Following Steps 1 and 2, the list of chemicals remaining is:

- Arsenic
- Antimony
- Lead
- Thallium

Antimony may be eliminated because the magnitude of the risk which it poses is very small compared to that posed by arsenic. For example, in the 10 samples most contaminated with arsenic, the average concentration of antimony is about 1% that of arsenic. Because the chronic oral RfD for antimony (4E-04) is slightly larger that the RfD for arsenic (3E-04), it is clear that the average non-cancer risk contributed by antimony is less than 1% of that contributed by arsenic. That is, if antimony was retained and the non-cancer risk were quantified, the risk would be less than 1% larger than if antimony were not included. Because an increment of 1% is well within the uncertainty range of the risk assessment procedure, inclusion of antimony would not change any risk interpretations and therefore is judged to be unnecessary.

Step 4. Special Investigation for Thallium

Data on thallium available from the existing TAL analyses are internally inconsistent, as shown below:

Parameter	Data Set 1	Data Set 2
Mean (ppm)	13.5	0.45
Max (ppm)	19	0.68
Detection Limit (ppm)	10	0.1

The basis for this internal inconsistency is not clear. One possibility is that differences in analytical methods are responsible. Data in Set 1 (collected during Phase I) utilized an analytical method (ICP-Trace, USEPA Method 6010) that had a relatively high detection limit, and most of the reported values were near that detection limit. In the second data set (specifically targeting samples high in arsenic), thallium was analyzed by USEPA Method 6020 (ICP-MS) which has a much lower detection limit for thallium. In general, the results of the second analysis are thought to be more reliable, and are in accord with expected thallium levels in background soils. However, because it is not certain that the results from the second analysis are actually more reliable than from the first, a special study was performed in which thallium levels were measured in six samples from each of data Set 1 and data Set 2. The details of this study are presented in Attachment 1 to this Appendix.

SUMMARY: CHEMICALS SELECTED AS COPCs AT VBI70

Based on the methods and data detailed above, the COPCs selected for quantitative evaluation at the VBI70 site are:

ARSENIC LEAD

Reference:

URS Operating Services [UOS]. 1998. Final Sampling Activities Report for North Denver Residential Soils – Phase I. June 1998.

USEPA. 1989. Risk Assessment Guidance for Superfund. Volume I. Human health Evaluation Manual (Part A). U.S Environmental Protection Agency, Office of Emergency and Remedial Response. EPA/540/1-89/002.

USEPA. 1999. U.S. Environmental Protection Agency. Region III Risk-Based Concentration Table. Philadelphia, PA. April 12.

TABLE 1 SUMMARY STATISTICS FOR SOIL SAMPLES

	•	Detection	Summary Statistics		
Analyte	N	Frequency	Min	Max	Mean
ALUMINUM	54	100%	4900	15000	8761
ANTIMONY	54	22%	2.2	54	6.8
ARSENIC	54	93%	5	9940	543
BARIUM	54	100%	91	1000	251
BERYLLIUM	54	98%	0.3	1.1	0.7
CADMIUM	54	100%	0.9	19	5.9
CHROMIUM	54	100%	7.2	99	22
COBALT	54	98%	1.0	7.0	4.6
COPPER (a)	53	100%	12	71	37
LEAD	54	100%	36	3550	712
MANGANESE	54	100%	160	560	323
MERCURY	54	93%	0.1	11	1.0
NICKEL	54	100%	5.9	96	11
SELENIUM	54	19%	0.3	10	9
SILVER	54	69%	0.3	3	0.7
THALLIUM	54	89%	0.2	19	11
VANADIUM	54	100%	13	42	21
ZINC	54	100%	84	3680	499
CALCIUM	54	100%	1900	41000	6757
IRON	54	100%	7900	26000	13405
MAGNESIUM	54	100%	1400	4100	2400
POTASSIUM	54	100%	1400	4100	2350
SODIUM	54	5%	300	440	304

a. Excludes one value (14,000 ppm) that is considered anomalous

TABLE 2 COMPARISON OF MAXIMUM VALUES TO RBCs

	Maximum	Region III	Retain as
Analyte	Conc (ppm)	RBC (ppm)	COPC?
ALUMINUM	15000	78400	no
ANTIMONY	54	31	yes
ARSENIC	9940	0.43	yes
BARIUM	1000	5500	no
BERYLLIUM	1.1	160	no
CADMIUM	19	78	no
CHROMIUM	99	230	no
COBALT	7.0	4700	no
COPPER (a)	71	3100	no
LEAD	3550	400	yes
MANGANESE	560	1600	no
MERCURY	11	23	no
NICKEL	96	1600	no
SELENIUM	10	390	no
SILVER	3	390	no
THALLIUM	19	5.5	yes
VANADIUM	42	550	no
ZINC	3680	23000	no
CALCIUM	41000		no
IRON	26000	23000	yes
MAGNESIUM	4100		no
POTASSIUM	4100		no
SODIUM	440		no

ATTACHMENT 1 SPECIAL STUDY ON THALLIUM IN SITE SOILS

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MEMO

To: Bonnie Lavelle, Chris Weis From: Bill Brattin, Mary Goldade

Date: 6/15/99

Subj: Special Evaluation of Thallium in VBI70 Soils

As we discussed, available data are internally inconsistent on the level of thallium (Tl) in site soils at VBI70. We suspect, but cannot demonstrate unambiguously, that levels are low (< 1 ppm), and are not of health concern. In order to resolve this issue, we recommend proceeding with a small study, as described below.

DATA QUALITY OBJECTIVES

State the Problem

Available data are not internally consistent regarding the level of thallium. One data set (measured by ICP-trace) suggests thallium levels are 10-20 ppm (above a level of concern). Another data set (measured by ICP-MS) suggests thallium exists at levels less than 1 ppm (below a level of concern). The problem is to determine which data set is correct.

Decisions to Be Made

The decision to be made is whether or not the Phase 3 soil sampling project will identify thallium as a contaminant of potential concern (COPC) and, therefore included on the target analyte list.

Types of Input Needed

Data required to resolve this issue are accurate and reliable measurements of thallium in site soil. This includes adequate quality assurance/quality control (QA/QC) data to establish that the measurements are reliable.

Decision Rule

If site soils contain thallium at levels largely or entirely below the Region III risk-based soil screening level (5 ppm), thallium may be dropped as a COPC. If site levels are largely or entirely above 5 ppm, thallium will be retained as a COPC.

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STUDY DESIGN

Number of samples

A total of 10 site soil samples will be analyzed for thallium. The 10 site soils will be comprised of 6 samples from the set of soils previously analyzed by ICP-trace (yielding Tl levels of 10-20 ppm) plus 4 samples from the set of soils previously analyzed by ICP-MS (yielding Tl levels below 1 ppm). It should be noted that different fractions of soils were analyzed for each method. During the past investigations (Phase I [UOS 1998a] and RBS [ISSI 1999a]), samples that were analyzed by ICP-trace were sieved to <2 mm prior to analysis, while samples analyzed by ICP-MS were sieved to $<250~\mu m$. Due to limited availability of archived samples, bulk samples (sieved to <2 mm) will be analyzed by ICP-trace, while raw samples (not sieved) will be analyzed by ICP-MS for this thallium study. Fine samples (sieved to $<250~\mu m$) were not available for analyses.

The list of specific samples selected and the analytical results for thallium available prior to this study are summarized in Table 1.

QA/QC

Four blind QC samples (certified reference materials) will be included in random order with the 10 site samples. These four samples will consist of two samples each of two commercial standards, including 1) a certified clean soil (low thallium, about 0.49 ppm), and 2) a certified thallium standard of approximately 5.0 ppm.

Analytical methods

Each sample will be analyzed for thallium by three analytical methods:

- ICP-trace (EPA SW-846 Method 6010B)
- ICP-MS (EPA SW-846 Method 6020)
- Graphite furnace (EPA SW-846 Method 7841)

Validation of Recovery

In order to assess the recovery of thallium from each sample, every sample will be analyzed in its original form along with a matrix spike (MS). The MS will be spiked so that the increment in thallium concentration is about 5 ppm.

TABLE 1. SAMPLE ID AND PREVIOUS THALLIUM RESULTS

Field Sample ID	CLP Lab Sample ID	XRF Lab Sample ID	Original Method ^a	Original Value Thallium (ppm)
C4690CYB-064	D9D100118-002	ND-98-782	ICP-MS	0.63
C4690CYB-046E	D9D100118-004	ND-98-640	ICP-MS	0.20
C4711THF-001	D9D100118-006	ND-98-549	ICP-MS	0.33
C4771VIN-001	D9D100118-0010	ND-98-245	ICP-MS	0.33
D4701JOS10	9804190-35	ND-98-016	ICP-T	10
D4145FIB10	9804190-19	ND-98-022	ICP-T	12
D4715GYF10	9804190-36	ND-98-027	ICP-T	17
D4050FIB10	9804190-41	ND-98-058	ICP-T	11
D4785CLF10	9804190-43	ND-98-064	ICP-T	15
D4780CBB10	9804190-26	ND-98-069	ICP-T	16

 $^{^{\}rm a}$ Samples analyzed by ICP-MS were sieved to <250 $\mu{\rm m}.$ Samples analyzed by ICP-T were sieved to <2 mm.

TABLE 2 LIST OF SAMPLES AND SAMPLE NUMBERS

Index	Field ID	Sample Number
1	Certified 0.49 ppm	Tl-15
	(RM 8604)	
2	Certified 0.49 ppm	Tl-3
	(RM 8604)	
3	Certified 5.0 ppm	TI-12
	(RM 8607)	
4	Certified 5.0 ppm	Tl-4
	(RM 8607)	, and the second
5	ND-98-016	T1-8
6	ND-98-022	T1-2
7	ND-98-027	Tl-6
8	ND-98-058	Tl-7
9	ND-98-064	Tl-11
10	ND-98-069	Tl-9
11	ND-98-245	Tl-16
12	ND-98-782	Tl-1
13	ND-98-640	T1-5
14	ND-98-549	Tl-10

	Appen	dix B:		
Site Map Depi	cting Concentra	tions of Arse	nic Across VBI	70

EPA REGION VIII SUPERFUND DOCUMENT MANAGEMENT SYSTEM

DOC ID #_	211	496	
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Appendix C:	
Phase III Investigation - Rationale for Collecti	ng Surface Soil Samples Only

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MEMORANDUM



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To:

Chris Weis & Bonnie Lavelle

From:

Mary Goldade

Date:

June 18, 1999

Project Name:

Vasquez Boulevard & I-70

RE:

Phase 3 Investigation - Rationale for Collecting Surface Soil Samples Only

cc:

Project Files

Purpose

Using soil data gathered from the Vasquez Boulevard and Interstate 70 (VBI70) site to date, determine whether the levels of arsenic and lead found in the surface soils (0-2 inches) are generally higher or lower than that of soils at depths of 6-10 inches. The conclusions reached in this memorandum will be the scientific basis for deciding whether soils at depth must included in the VBI70 Phase 3 study design.

Historical Data

Paired surface soil and depth samples were collected in three previous sampling programs: Phase I/II (UOS 1998a, 1998b); and Risk-Based Sampling (ISSI 1999a). The primary objective for each of these investigations was to determine the nature and extent of arsenic and lead levels in soils at the VBI70 site. Therefore, this investigation included collection of both surficial and depth samples.

Depth samples were collected for the Phase I/II sampling program at approximately 6-10 inches from the soil horizon at most residences from which surface soils were also collected. All soils were tested for both lead and arsenic. In general, the levels of lead and arsenic found in the surficial soils were at least as high and usually higher than those found in the corresponding depth sample.

As part of the Risk-Based Sampling (RBS) Program, the concentration profile for arsenic, lead, cadmium and zinc in residential soils were measured. This was done by collecting soil cores in the 0-12 inch horizon and measuring target metals (arsenic, lead, cadmium and zinc) in each 2 inch interval. These data are presented in the Draft Report for the VBI70 Residential Risk-Based Sampling Stage I Investigation (ISSI Consulting Group [ISSI] 1999a). As seen, although the absolute concentration of each metal varies considerably, concentrations of all target metals (arsenic, lead cadmium and zinc) tend to decrease with increasing depth.

Summary of Results

The historical data obtained for Phase I/II and the RBS were further evaluated and the results of these evaluations are provided in Table 1. In brief, the ratio of the metals levels present in surface soils to those found in the corresponding depth samples were calculated for each surface soil and depth sample pair. Finally, the mean surface to depth ratios were calculated for each of 5 metals concentration intervals for both the Phase I/II and RBS investigations. If the value of the ratios are equal to one, then the metals levels in surface soils are approximately equal those in the depth samples. If the ratios are greater than one, then the metals levels in the surficial soils are greater than those found at depth. Alternatively, if the ratios are less than one, then the metals levels in the depth samples are greater than those found in surface soils.

As seen in Table 1, arsenic and lead levels measured during Phase I/II have at minimum a 1:1 relationship for

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corresponding surface and depth samples and the arsenic and lead levels in surface soils increase as the surface:depth ratio increases. Therefore, the highest metals levels tend to occur at the surface. This general trend is also observed for the RBS data.

Conclusion

When contamination is present in soils below the 0-2 inch depth, contamination is generally also present in the corresponding surficial soils at equal or greater concentrations. Therefore, a study that is designed to identify residences with elevated levels of contaminants of potential concern (COPCs) in the surface soils will be sufficient in identifying the impacted locations. Phase 3 soil investigations at VBI70 will be restricted to collection of surface soils only. If levels of COPCs are determined to be present in the residential surface soils above a level of concern, the residence may be investigated further to determine if and to what depth remediation of yard soils is necessary.

Table 1 - Descriptive Statistics for Surface:Depth Ratios of Metals Levels

Measured for the Phase I & II and Risk-Based Sampling Events (Intensive Grid Samples)

	Phase I & II		Risk-Based Sampling Event (Intensive Grid Sampling)		
Mean Conc. at		Mean Surface:		Mean Surface:	
Surface (mg/kg)	N	Depth Ratio	N	Depth Ratio	
Arsenic			10000000000000000000000000000000000000		
<70	1043	1.0	4	0.4	
71-150	142	1.4	3	0.9	
151-450	60	3.0	4	1.9	
451-1000	18	5.9	2	4.0	
>1000	0	9.0	3	5.2	
Lead					
<400	1176	1.4	6	1.4	
401-1000	85	2.0	5	2.9	
1001-1500	4	3.0	1	2.5	
1500-2000	1	6.6	3	5.4	
>2000	0		1	3.9	
Cadmium					
<78	NT	NT	16	1.7	
78-156	NT	NT	0		
157-234	NT	NT	0		
235-312	NT	NT	0		
>312	NT	NT	0		
Zinc					
<23,000	NT	NT	16	1.8	
23,001-46,000	NT	NT	0		
46,001-69,000	NT	NT	0		
69,001-92,000	NT	NT	0		
>92,000	NT	NT	0		

⁻ Not Applicable

a -Surface soil (0-2" depth); Depth soils (~6-10" depth)

b - Intensive residences include focal and adjacent properties

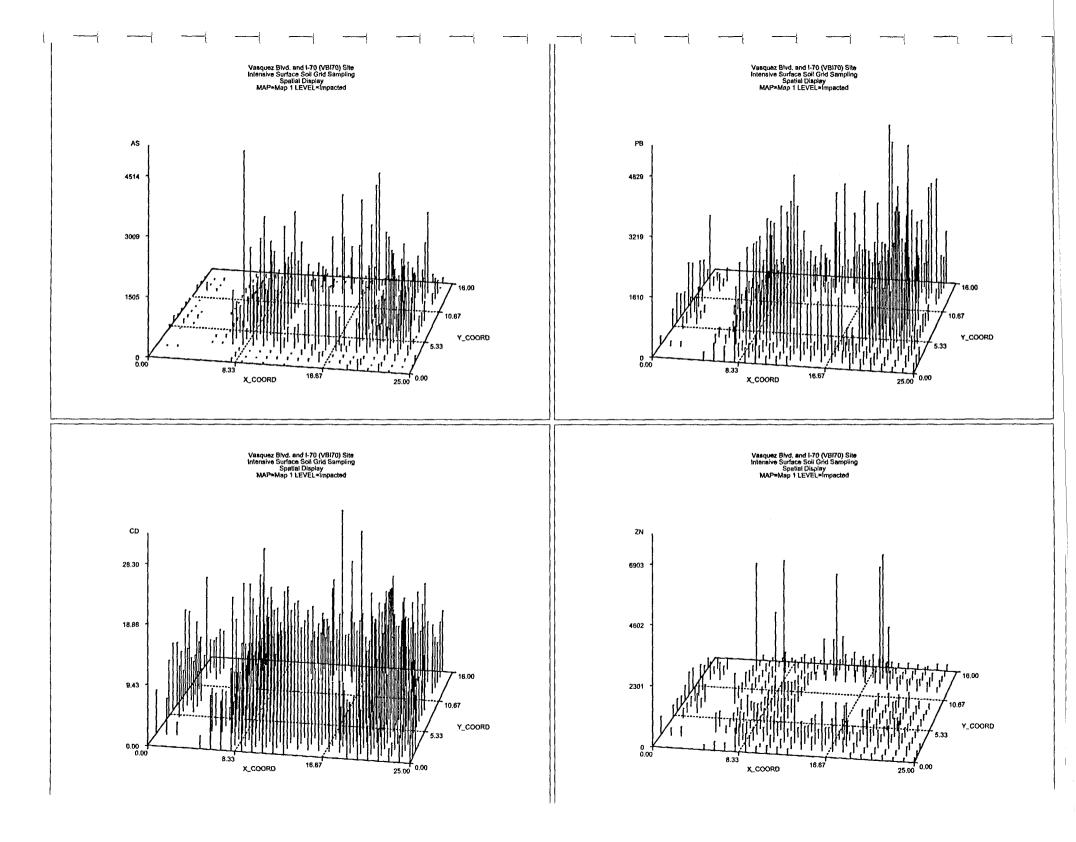
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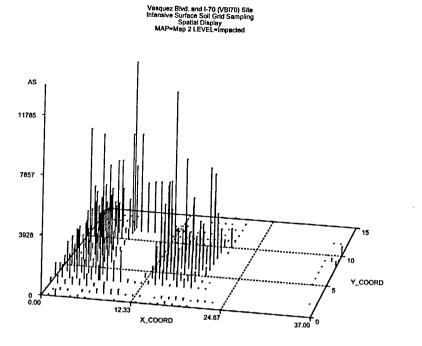
Appendix D:

Summary of Statistical Analysis

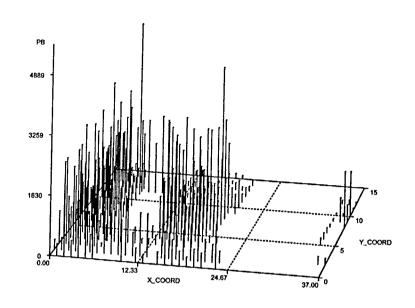
- Simulations performed on risk-based sampling data from locations 3,4, and 5.
- Simulations to determine the normality of composite samples using the minimum value as the reporting limit.
- Simulations to determine the normality of composite samples using estimated values below the reporting limit.

Spatial Display of Focal Properties

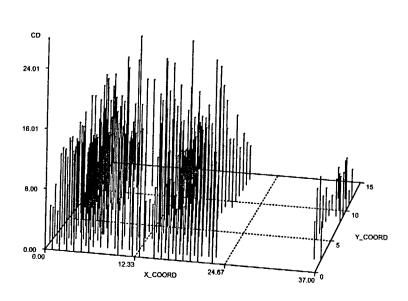




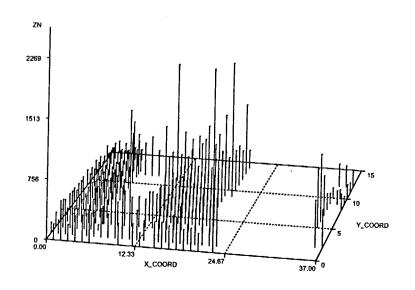


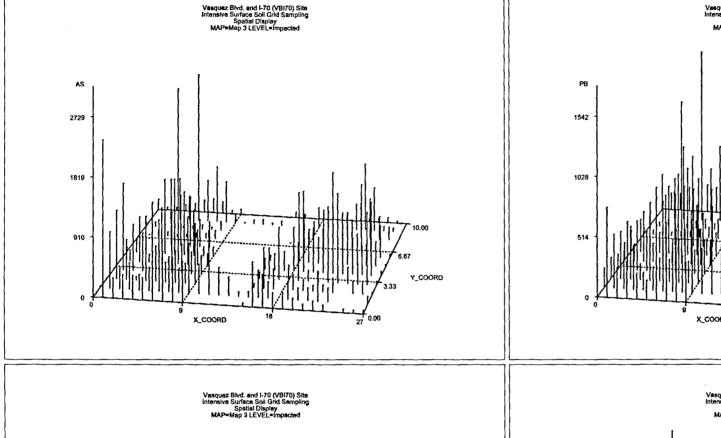


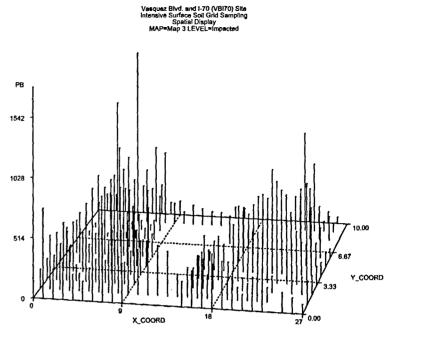
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Spatlal Display MAP=Map 2 LEVEL=Impacted

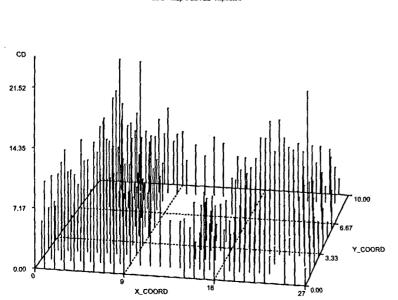


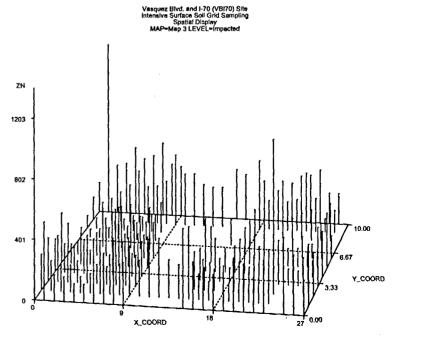
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Spatial Display MAP=Map 2 LEVEL=Impacted

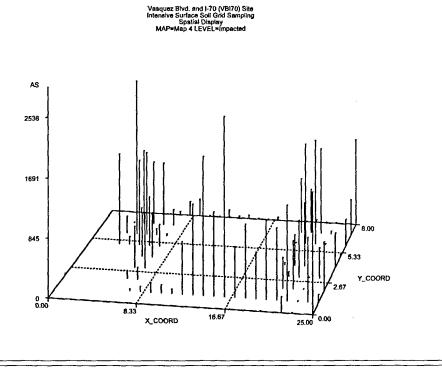


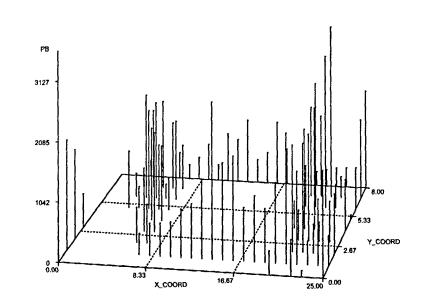






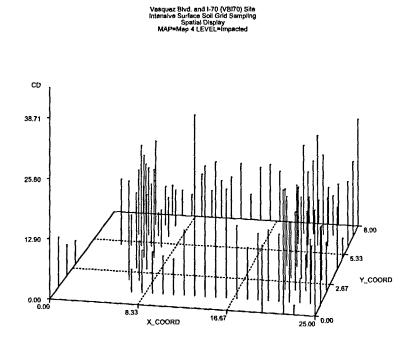


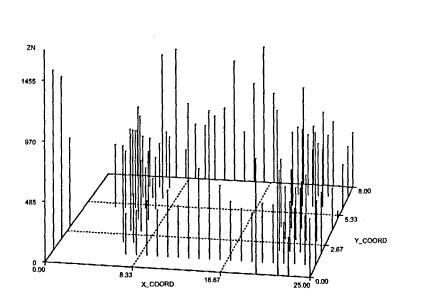


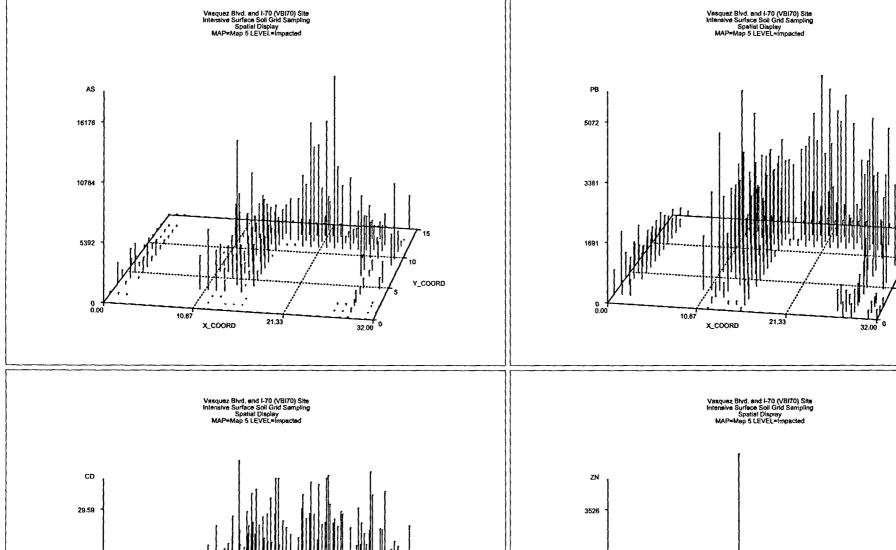


Vasquez Blvd. and 1-70 (VBI70) Site Intensive Surface Soil Grid Sampling Spatial Display MAP=Map 4 LEVEL=Impacted

Vasquez Bivd, and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Spatial Display MAP=Map 4 LEVEL±Impacted







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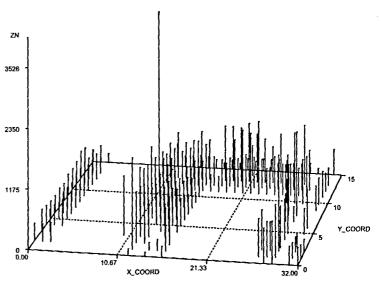
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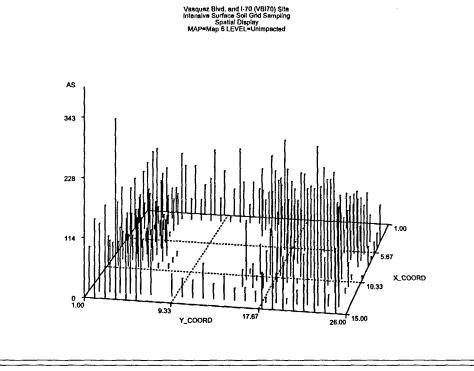
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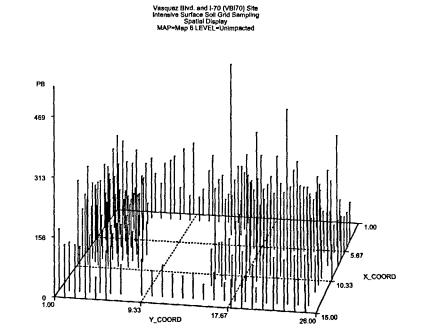
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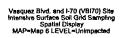
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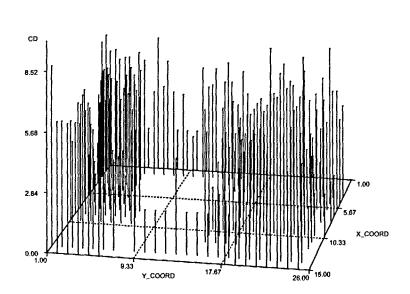


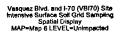
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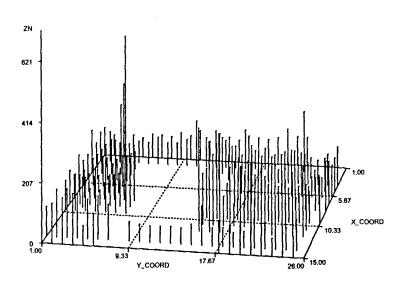


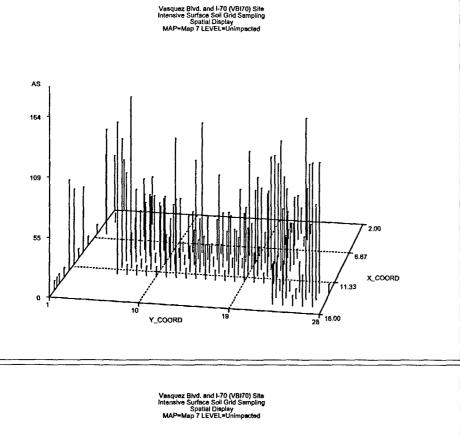


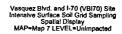


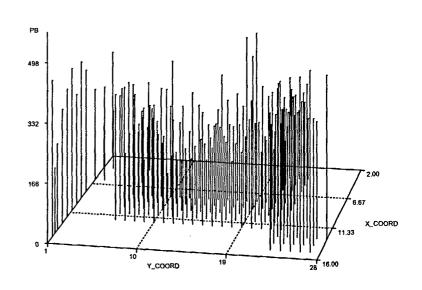


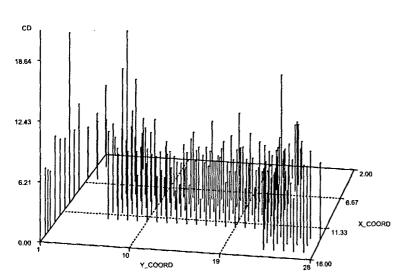




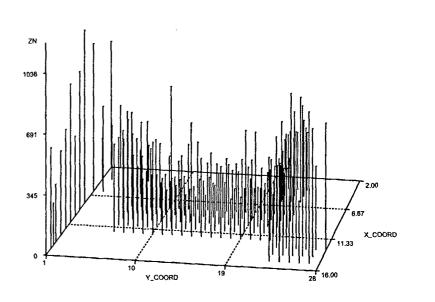


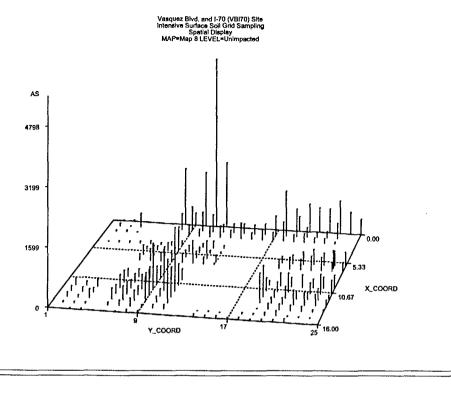


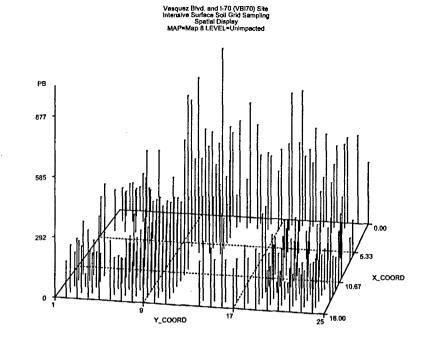


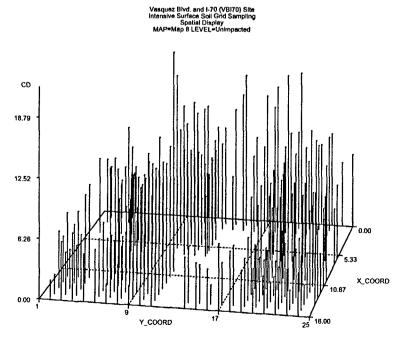


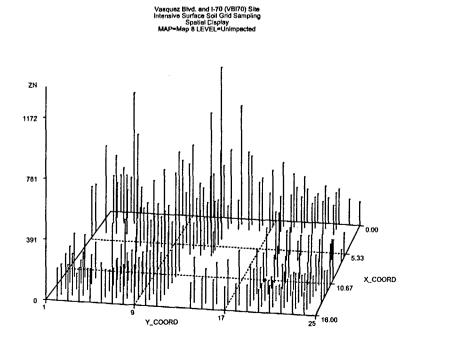
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Spatial Display MAP=Map 7 LEVEL=Unimpacted



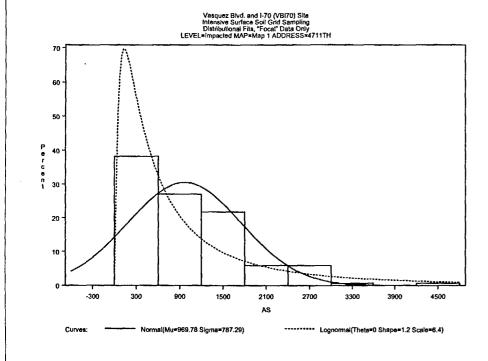


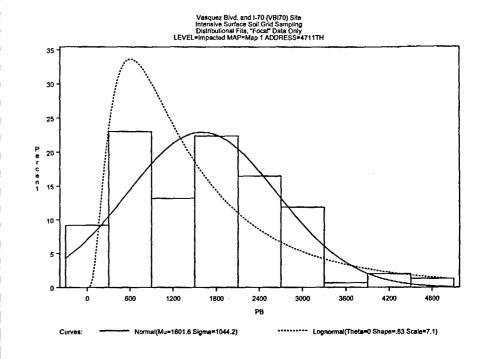


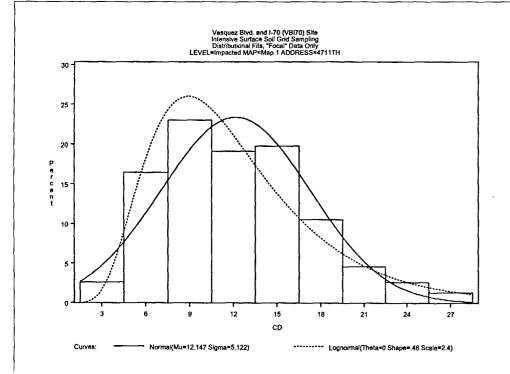


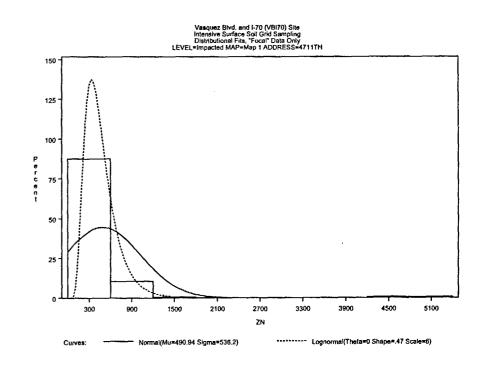


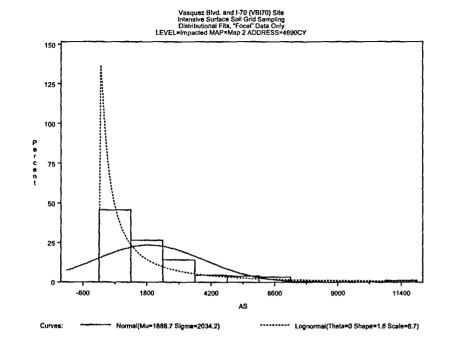
Simulations performed on risk-based sampling data from locations 3,4, and 5

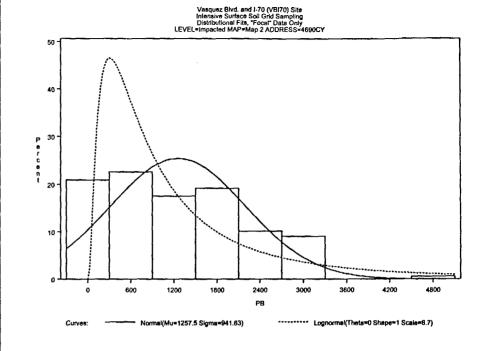


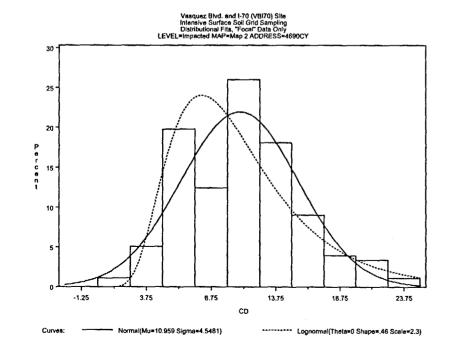


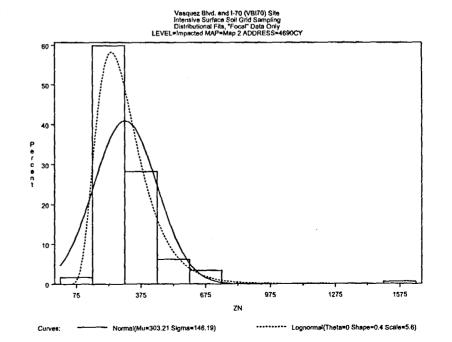


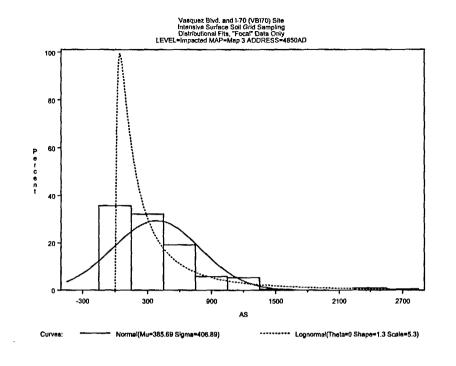


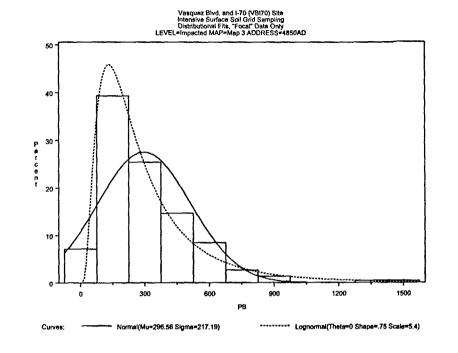


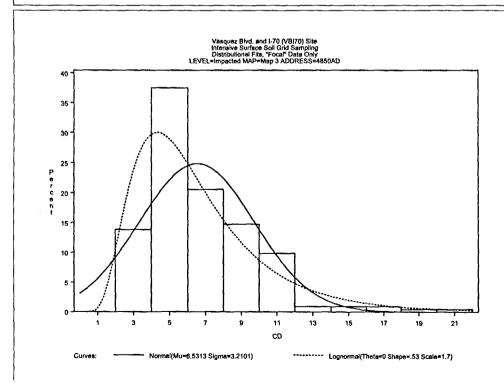


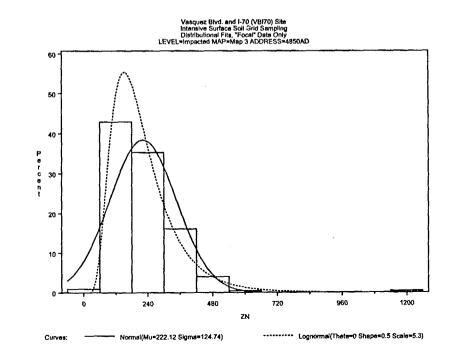


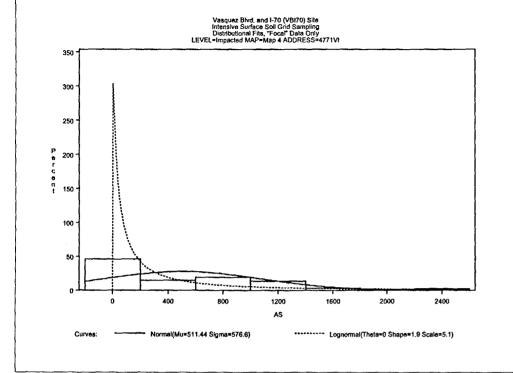


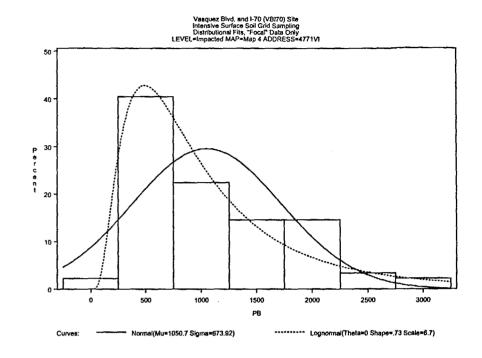


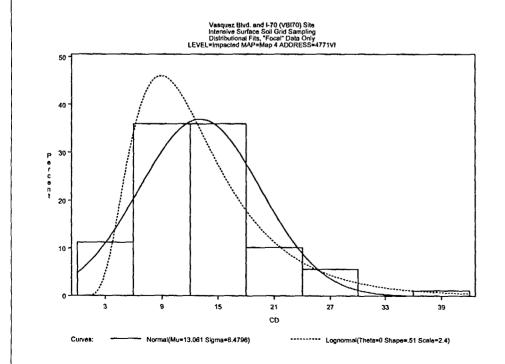


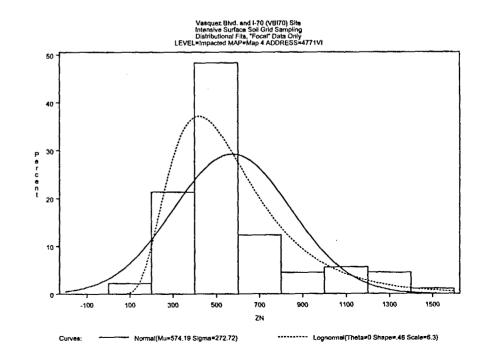


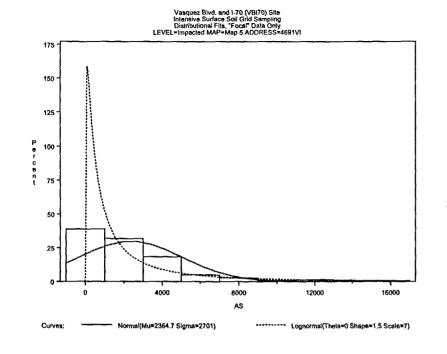


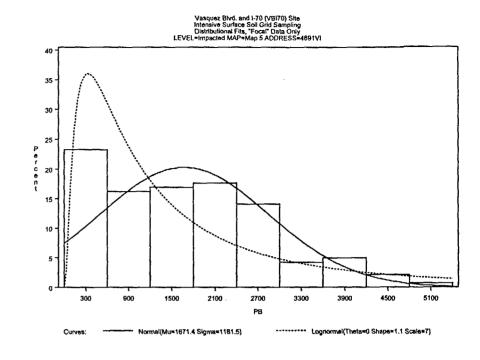


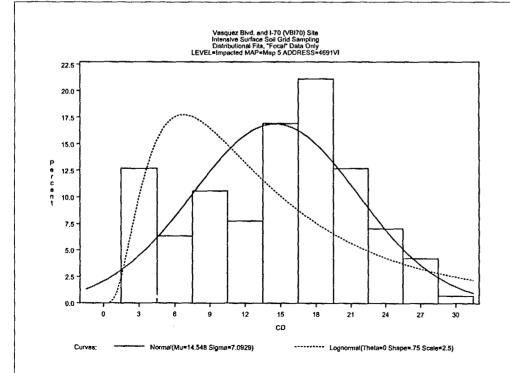


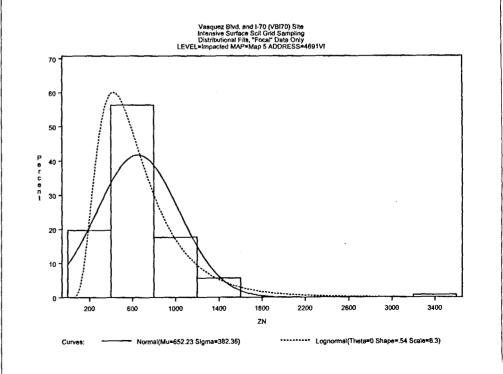


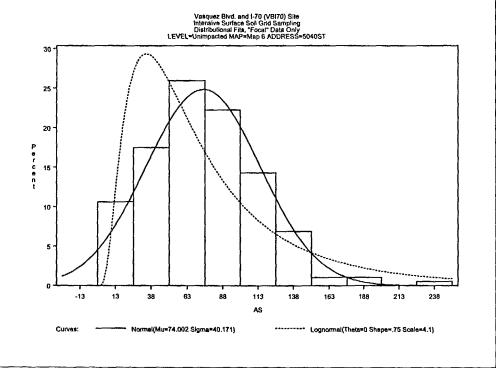


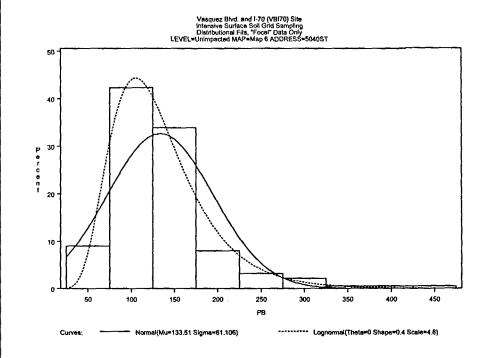


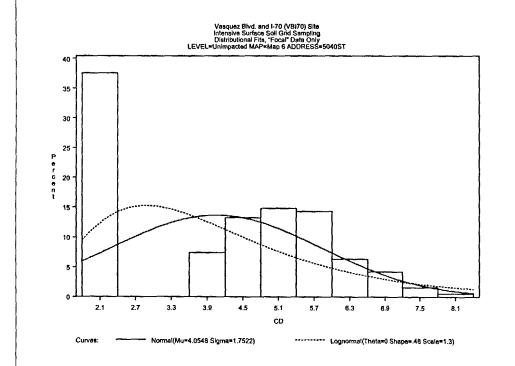


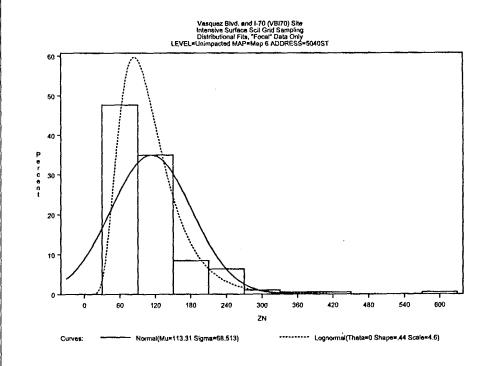


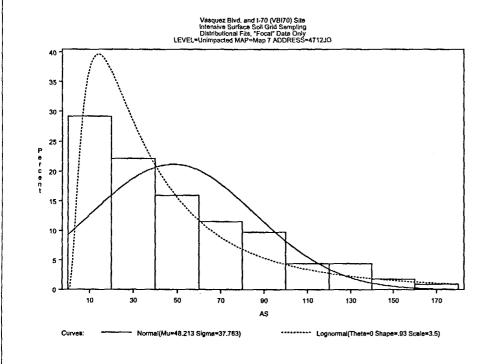


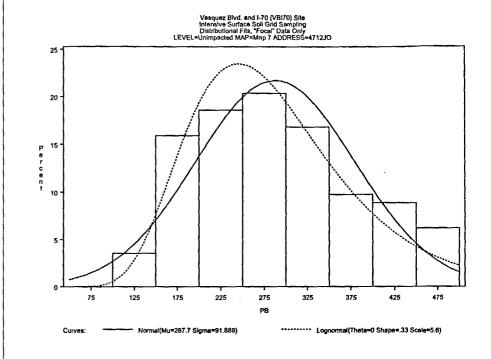


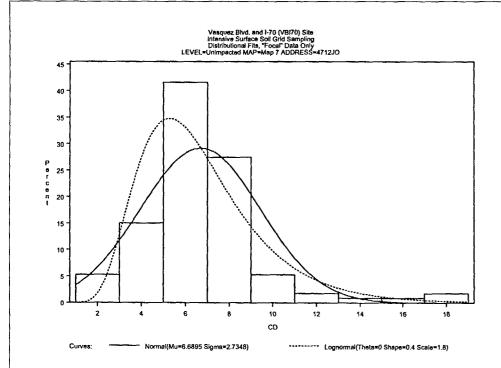


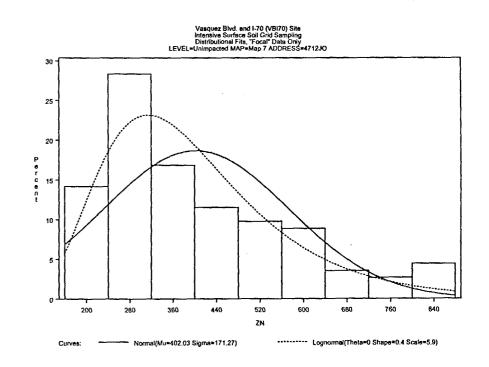


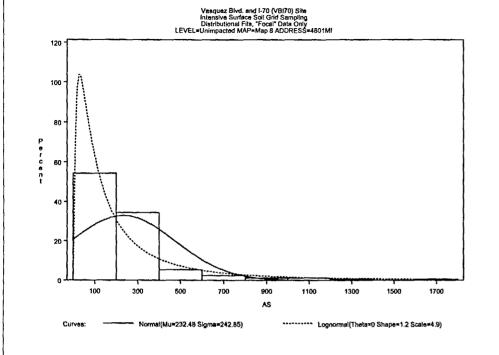


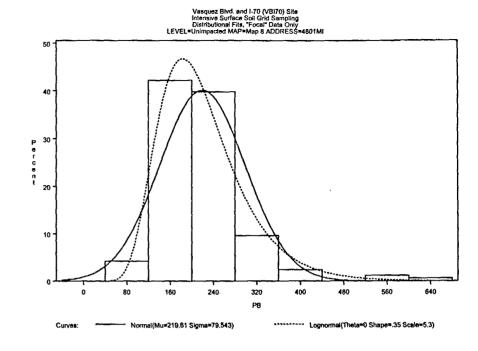


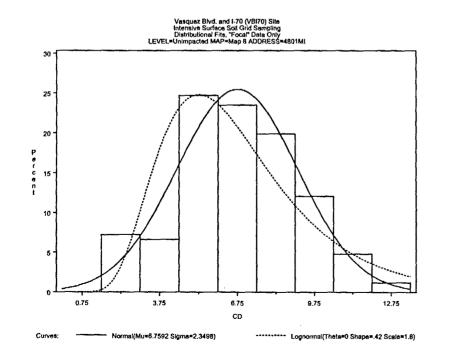


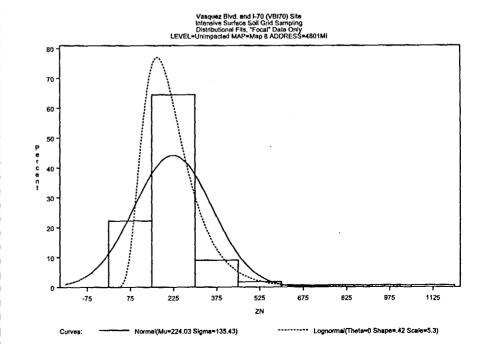










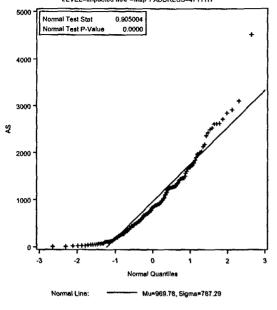


Vasquez	Boulevard	& I-70 -	Phase III
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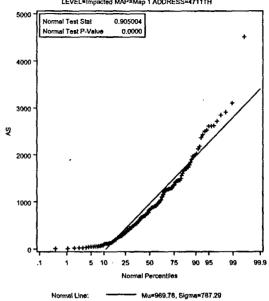
DRAFT- Do Not Cite

Simulations to determine the normality of composite samples using the minimum value as the reporting limit

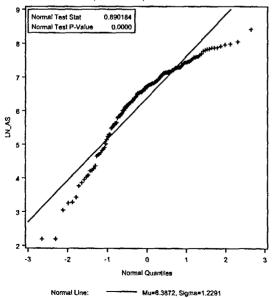
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Distributional Fits, "Focal" Data Only LEVEL=Impacted MAP=Map 1 ADDRESS=4711TH



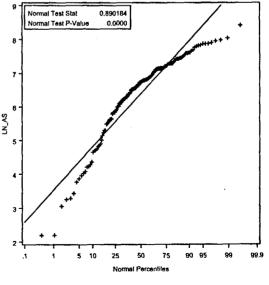
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Distributional Fits, "Focal" Data Only LEVEL=Impacted MAP=Map 1 ADDRESS=4711TH



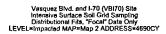
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Distributional Fis, "Focal" Data Only LEVEL=Impacted MAP=Map 1 ADDRESS=4711TH

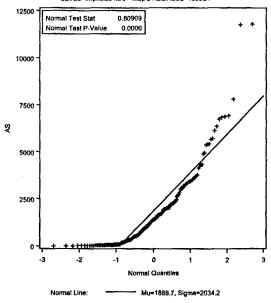


Vasquez Bivd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Distributional Fits, "Focel" Data Only LEVEL=Impacted MAP=Map 1 ADDRESS=4711TH

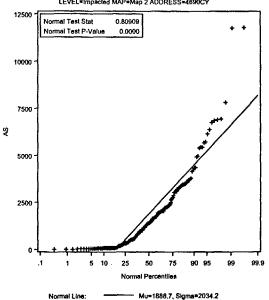


Normal Line: Mu=8.3872, Sigma=1.2291

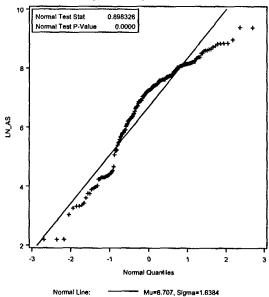




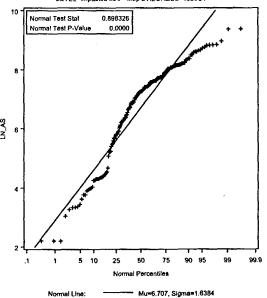
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Distributional Fits, "Focal" Data Only LEVEL=Impacted MAP≖Map 2 ADDRESS≃4690CY

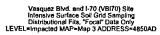


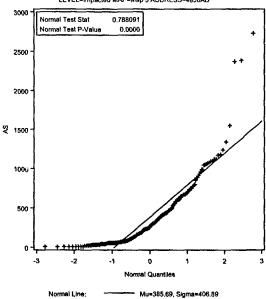
Vasquez Bivd, and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Distributional Fits, "Focal" Data Only LEVEL=Impacted MAP=Map 2 ADDRESS=4690CY



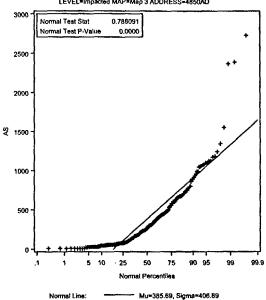
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Distributional Fits, "Focal" Data Only LEVEL=Impacted MAP=Map 2 ADDRESS=4690CY



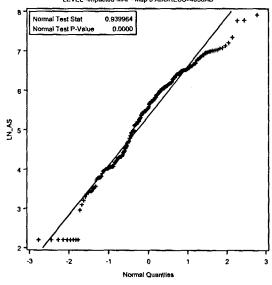




Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Distributional Fils, "Focat" Data Only LEVEL=Impacted MAP=Map 3 ADDRESS=4850AD

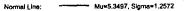


Vasquez Blvd, and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Distributional Fits, "Foca" Data Only LEVEL=Impacted MAP=Map 3 ADDRESS=4850AD

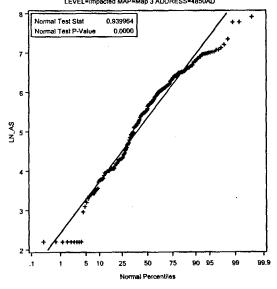


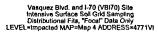
Mu=5.3497, Sigma=1.2572

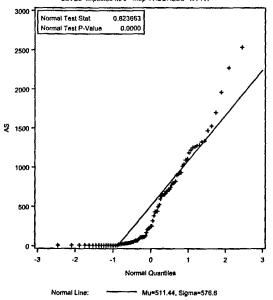
Normal Line:



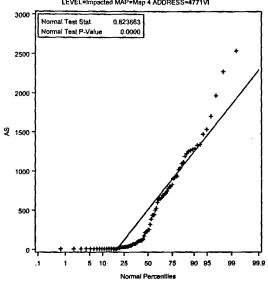
Vasquez Blvd, and I-70 (VB170) Site Intensive Surface Soii Grid Sampling Distributional Fits, "Focal" Data Only LEVEL=Impacted MAP=Map 3 ADDRESS=4850AD





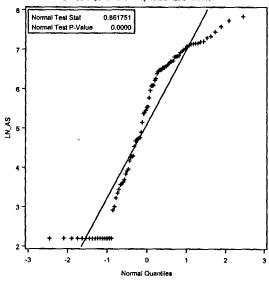


Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Distributional Fits, "Focal" Data Only LEVEL=Impocted MAP=Map 4 ADDRESS=4771VI



Normal Line: Mu=511.44, Sigma=576.6

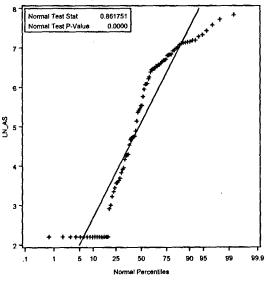
Vasquez Blvd. and I-70 (VB(70) Site Intensive Surface Soil Grid Sampling Distributions Fits, "Foce!" Data Only LEVEL=Impacted MAP=Map 4 ADDRESS=4771VI



Mu=5.0979, Sigma=1.8813

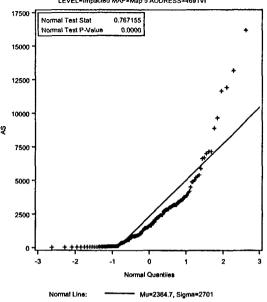
Normai Line:

Vasquez Blvd. and 1-70 (VB)70) Site Intensive Surface Goll Grid Sampling Distributional Fits, "Focal" Data Only LEVEL=Impacted MAP=Map 4 ADDRESS=4771VI

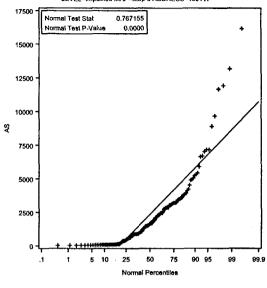


Normal Line: Mu=5.0979, Sigma=1.8813

Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soll Grid Sampling Distributional Fits, "Focal" Data Only LEVEL=Impacted MAP≖Map 5 ADDRESS≃4691VI

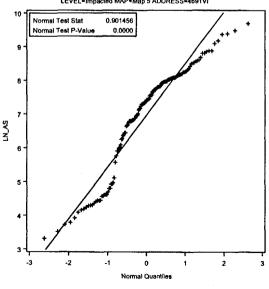


Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soll Grid Sampling Distributional Fits, "Focal" Data Only LEVEL=impacted MAP≭Map 5 ADDRESS≭4691VI



Normal Line: Mu=2364.7, Sigma=2701

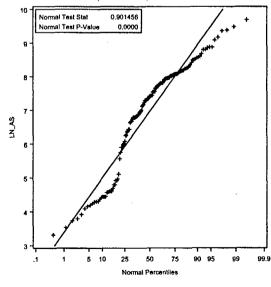
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soll Grid Sampling Distributional Fits, "Focal" Data Only LEVEL=Impacted MAP=Map 5 ADDRESS=4691VI



Mu=6.9719, Sigma=1.5376

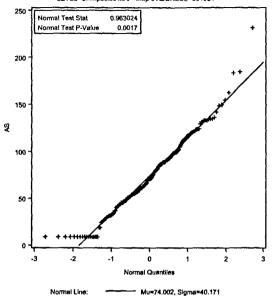
Normal Line:

Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Distributional Fits, "Focal" Data Only LEVEL=Impacted MAP=Map 5 ADDRESS=4691VI

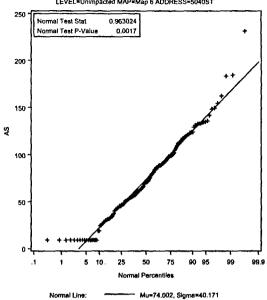


Normal Line: Mu=6.9719, Sigma=1.5376

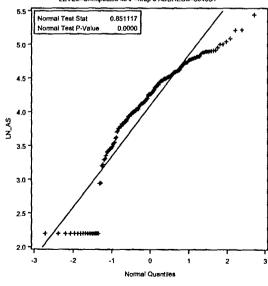




Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Distributional Fits, "Focal" Data Only LEVEL=Unimpacted MAP=Map 6 ADDRESS=5040ST



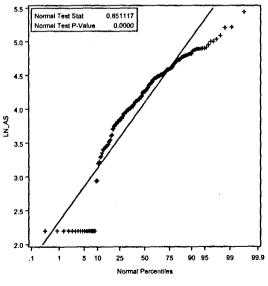
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Distributional Fits, "Focal" Data Only LEVEL=Unimpacted MAP=Map 8 ADDRESS=5040ST



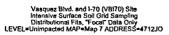
Mu=4.0935, Sigma=0.7524

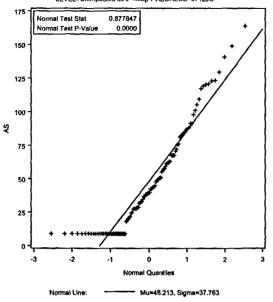
Normal Line:

Vasquez Blvd. and i-70 (VBI70) Site Intensive Surface Soll Grid Sampling Distributional Fits, "Focal" Data Only LEVEL=Unimpached MAP=Map 8 ADDRESS=5040ST

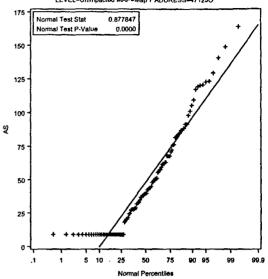


Normal Line: Mu=4.0935, Sigma=0.7524



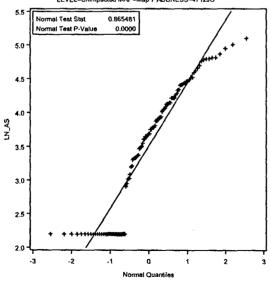


Vasquez Bivd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Distributional Fits, "Focat" Data Only LEVEL="Unimpacted MAP=Map 7 ADDRESS=4712JO



Normal Line: Mu=48.213, Sigma=37.763

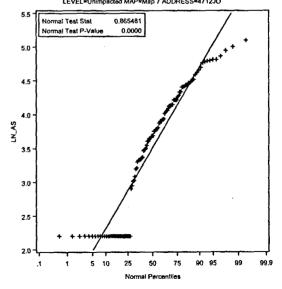
Vasquez Blvd. and I-70 (VB!70) Site Intensive Surface Soil Grid Sampling Distributional Fits, "Focal" Data Only LEVEL=Unimpacted MAP=Map 7 ADDRESS=4712JO



— Mu≠3.5061, Sigma≠0.9316

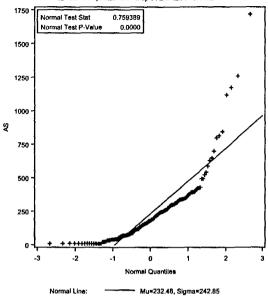
Normal Line:

Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Distributional Fits, "Focal" Data Only LEVEL=Unimpacted MAP=Map 7 ADDRESS=4712JO

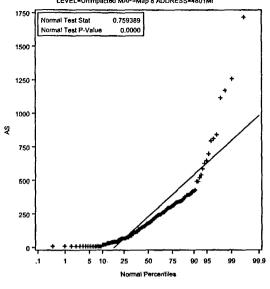


Normal Line: Mu=3.5061, Sigma=0.9316



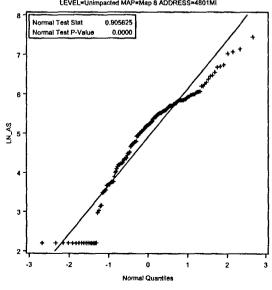


Vasquez Blvd, and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Distributional Fits, "Focal" Data Coly LEVEL=Unimpacted MAP=Map 8 ADDRESS=4801MI



Normal Line: Mu=232.48, Sigma=242.85

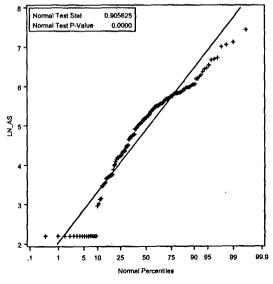
Vasquez 8vd. and I-70 (VBI70) Site Intensive Surface Soll Grid Sampling Distributional Fits, "Focal" Dats Only LEVEL≃Unimpacted MAP≃Map 8 ADDRESS≃4801MI



Mu=4.8913, Sigma=1.2296

Normal Line:

Vasquez Blvd. and 1-70 (VBi70) Site Intensive Surface 3oil Grid Sampling Distributional Fits, "Focal" Data Only LEVEL≃Unimpacted MAP=Map 8 ADDRESS=4801MI

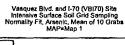


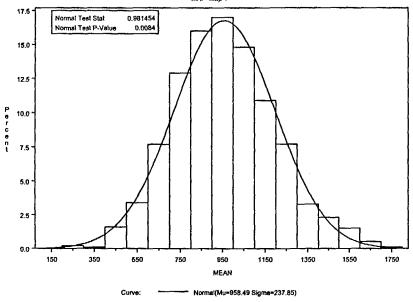
Normai Line: Mu=4.8913, Sigma=1.2296

Vasquez Bor	ılevard &	I-70 - I	Phase III
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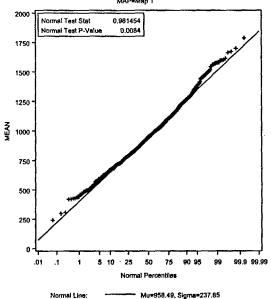
DRAFT- Do Not Cite

Simulations to determine the normality of composite samples using estimated values below the reporting limit

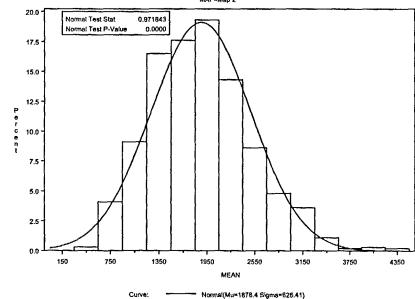




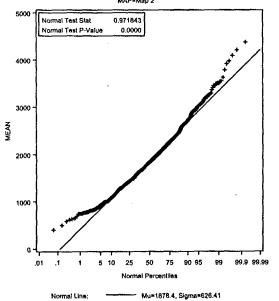
Vasquez Blvd. and F70 (VBI70) Site Intensive Surface Soil Grid Sampling Nomality Fit, Arsenic, Mean of 10 Grabs MAP=Map 1



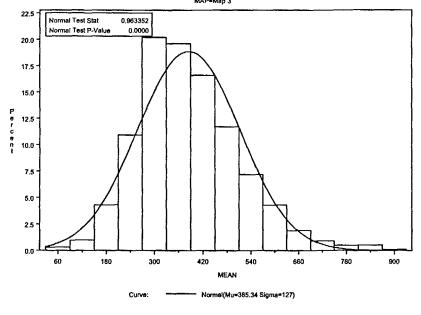




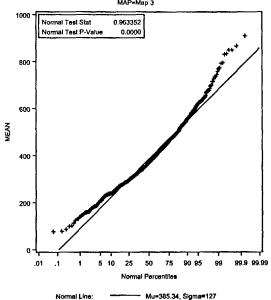
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soll Grid Sampling Normality Fit, Arsenic, Mean of 10 Grabs MAP=Map 2



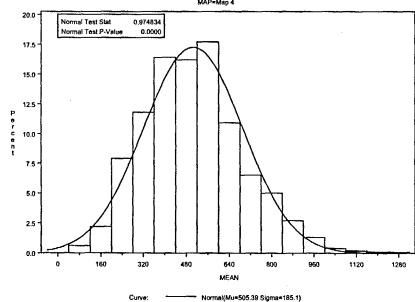




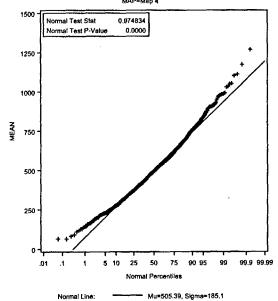


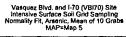


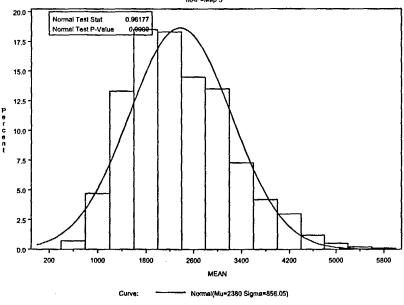
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 10 Grabs MAP=Map 4

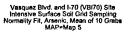


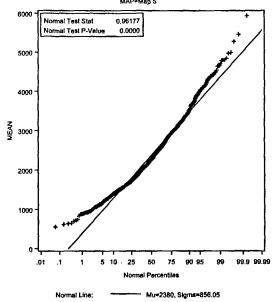
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Goll Grid Sampling Normality Fit, Arsenic, Mean of 10 Graba MAP=Map 4



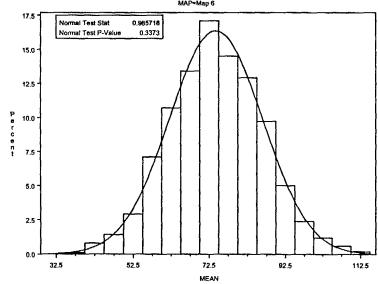








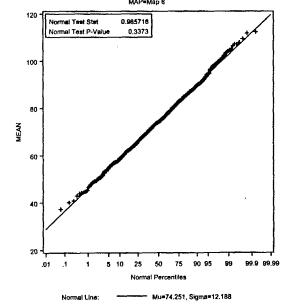
Vasquez Blvd. and I-70 (VBI70) Site tritensive Surface Soit Grid Sampling Normality Fit, Arsenic, Mean of 10 Grabs MAP≃Map 6



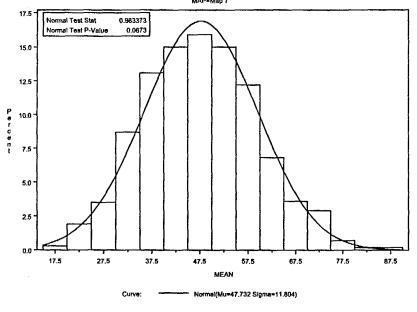
- Normal(Mu=74.251 Sigma=12.188)

Curve:

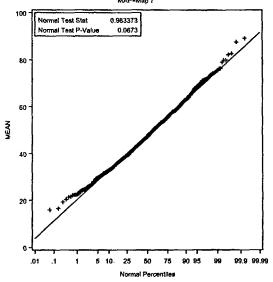
Vasquez Blvd. and I-70 (VBI70) Site intensive Surface Soit Grid Sampling Normality Fit, Arsenic, Mean of 10 Graba MAP=Map 6





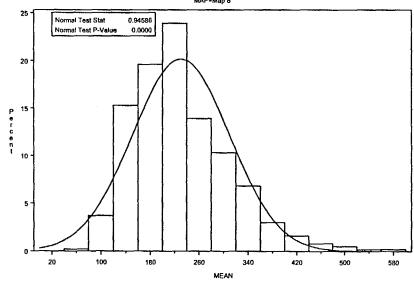


Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 10 Grabs MAP=Map 7



Normal Line: Mu=47.732, Sigma=11.804

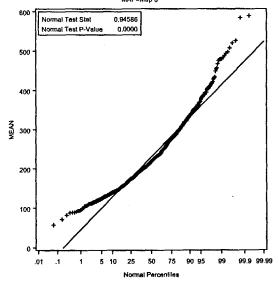
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grld Sampling Normality Fit, Arsenic, Mean of 10 Grabs MAP=Map 8



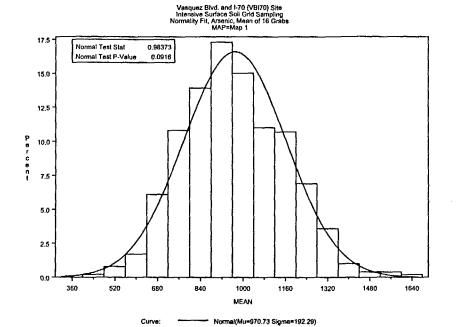
Normal(Mu=230.23 Sigma=79,159)

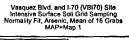
Curve:

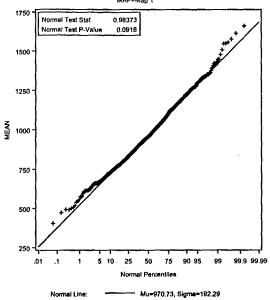
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Scil Grid Sampling Normality Fit, Arsenic, Mean of 10 Grabs MAP×Map 8

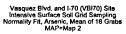


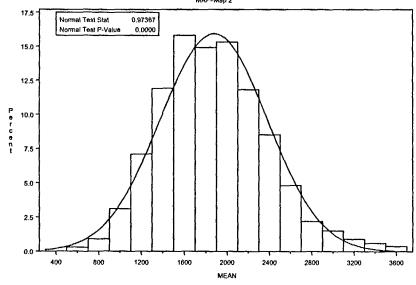
Normal Line: Mu=230.23, Sigma=79.159







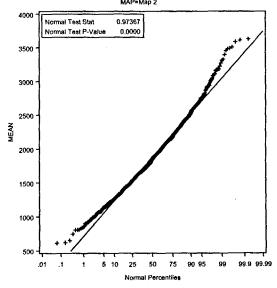




Curve:

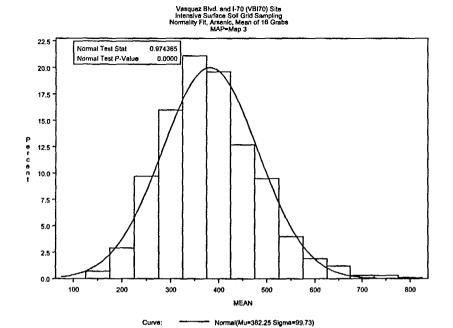
--- Normal(Mu=1877.1 Sigma=500.97)

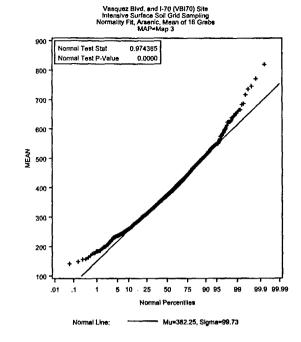
Vasquez Bivd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 16 Grabs MAP=Map 2

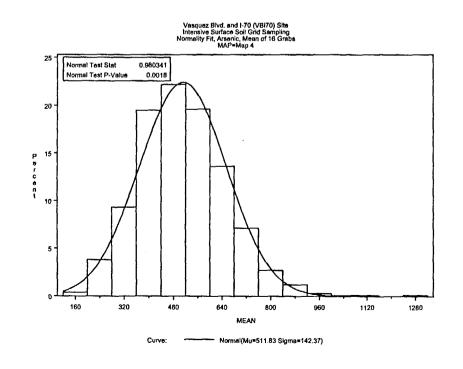


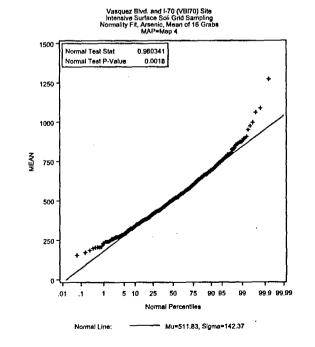
Mu=1877.1, Sigma=500.97

Normal Line:

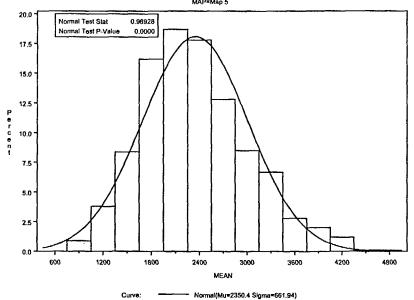




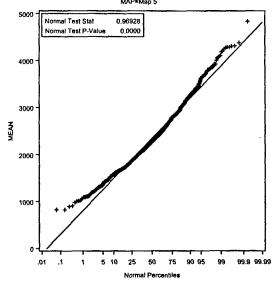






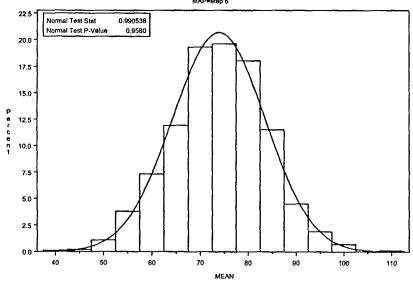


Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 16 Grabs MAP*Map 5



Normal Line: Mu=2350.4, Sigma=661.94

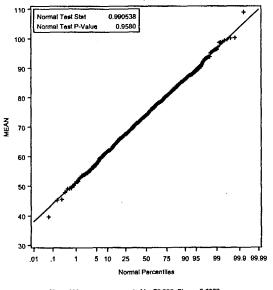
Vasquez Blvd. and I-70 (VB170) Site Intensive Surface Soll Grid Sampling Normality Fit, Arsenic, Mean of 16 Grabs MAP=Map 6



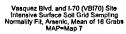
Normal(Mu=73.896 Sigma=9.6522)

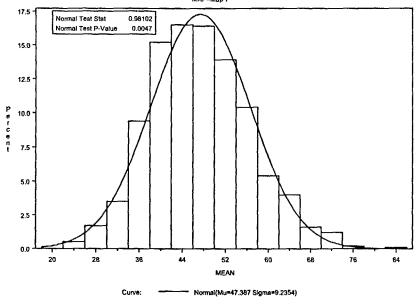
Curve:

Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 18 Grabs MAP=Map 6

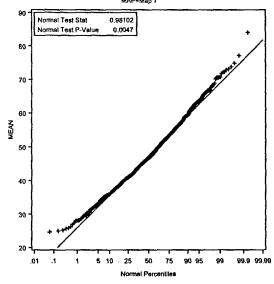


Normal Line: Mu=73.896, Sigma=9.6522



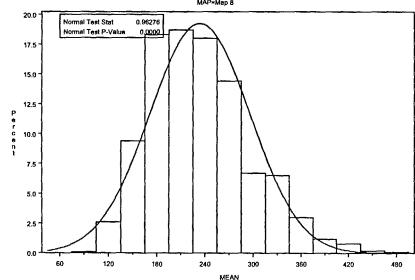


Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 16 Grabs MAP#Map 7



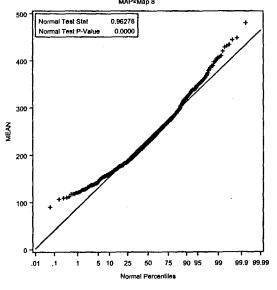
Normal Line: Mu=47.387, Sigma=9.2354

Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 16 Grabs MAP=Map 8

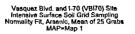


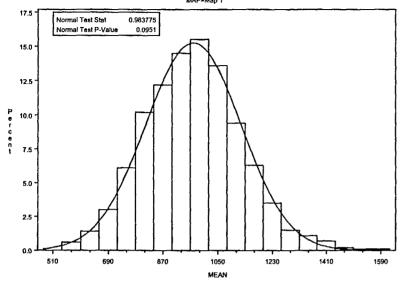
Curve: Normal(Mu=233.42 Sigma=62.207)

Vasquez Bivd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 16 Graba MAP=Map 8

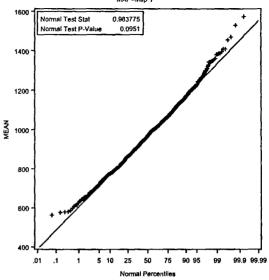


Normal Line: Mu=233.42, Sigma=62.207





Vasquez Blvd. and 1-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 25 Grabs MAP=Map 1

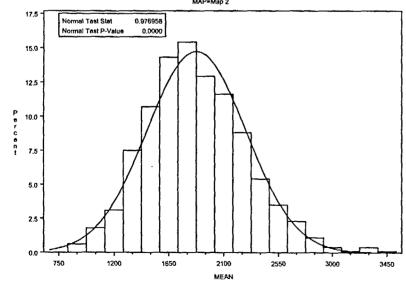


Normal Line; Mu=970.09, Sigma=158.83

Vasquez Blvd. and 1-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 25 Grabs MAP=Map 2

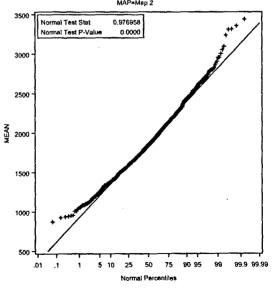
Curve:

--- Normal(Mu=970.09 Sigma=156,83)

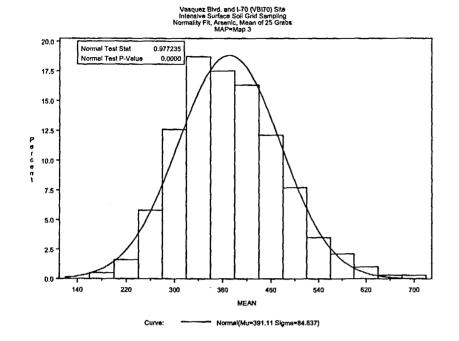


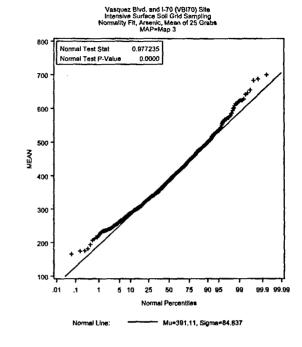
Curve: Normal(Mu=1875.7 Sigma=406.54)

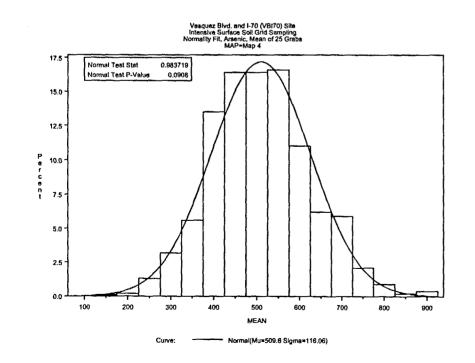
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 25 Grabs MAP=Map 2

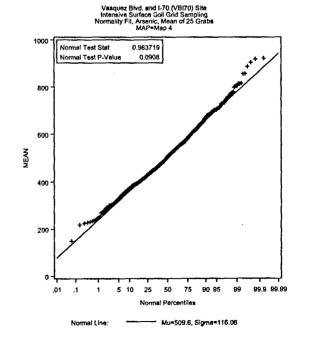


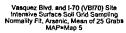
Normal Line: Mu=1875.7, Sigma=406.54

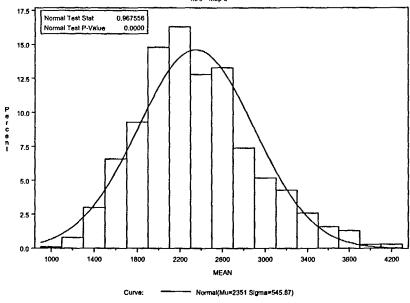




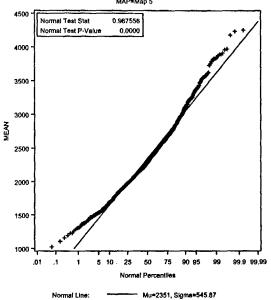




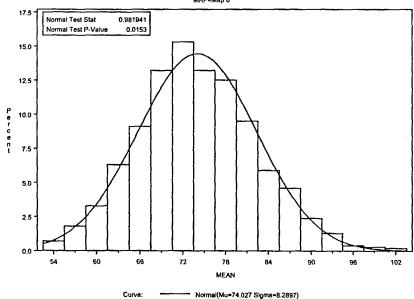




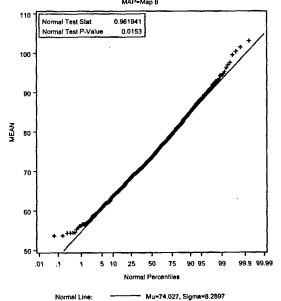
Vasquez Blvd, and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 25 Grabs MAP=Map 5

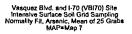


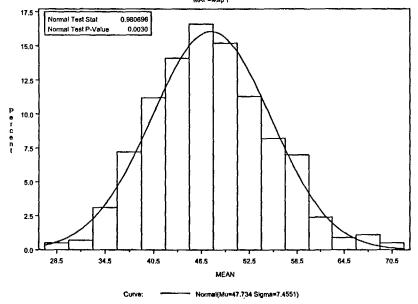
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 25 Grabs MAP≈Map 8



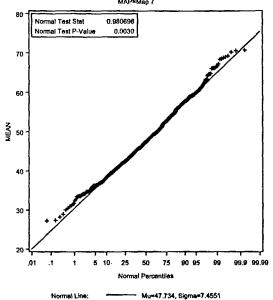
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 25 Grabs MAP=Map 6



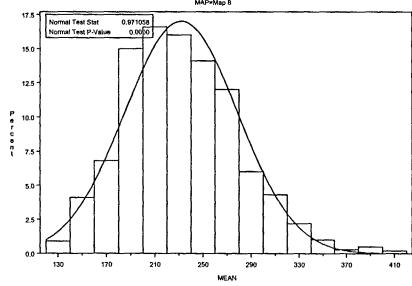




Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soli Grid Sampling Normality Fit, Arsenic, Mean of 25 Grabs MAP≃Map 7



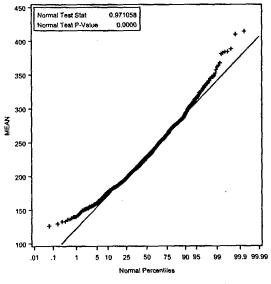
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 25 Graba MAP⇒Map B



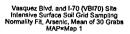
Curve:

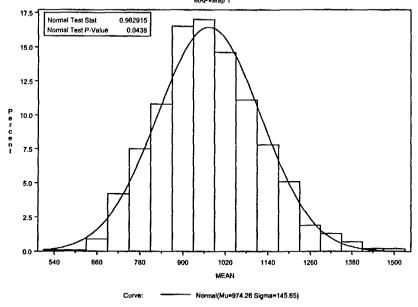
Normal(Mu=231.89 Sigma=46.911)

Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 25 Grabs MAP=Map 8

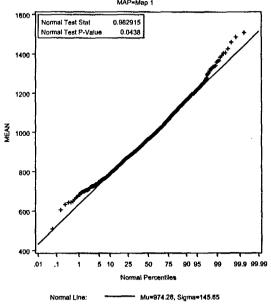


Normal Line: Mu=231.89, Sigma=46.911

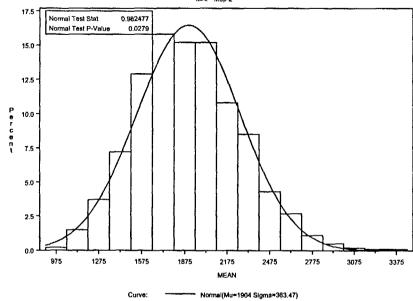




Vasquez Blvd. and 1-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 30 Grabs MAP=Map 1

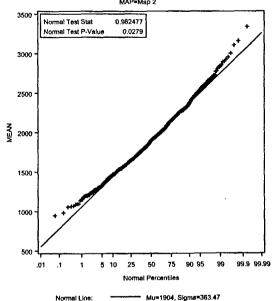


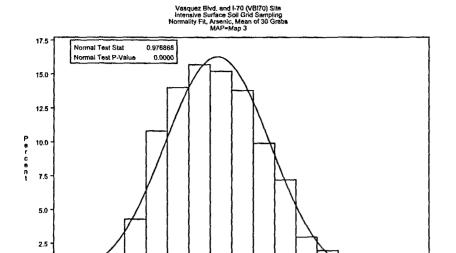
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 30 Grabs MAP=Map 2

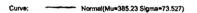


Curve:

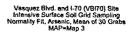
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface 3oil Grid Sampling Normality Fit, Arsenic, Mean of 30 Grabs MAP=Map 2

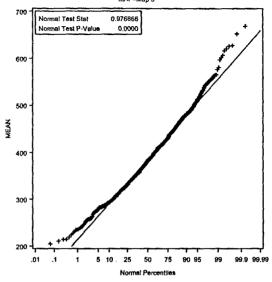




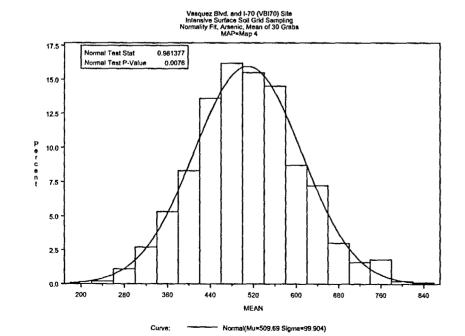


MEAN

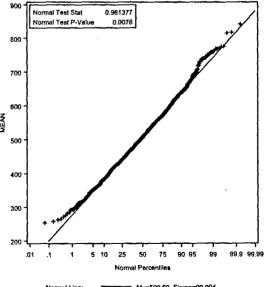




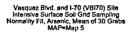


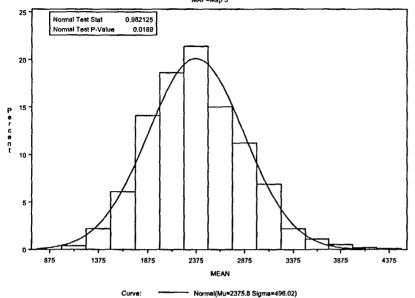




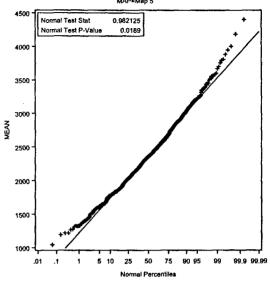


Normal Line: Mu=509.69, Sigma=99.904



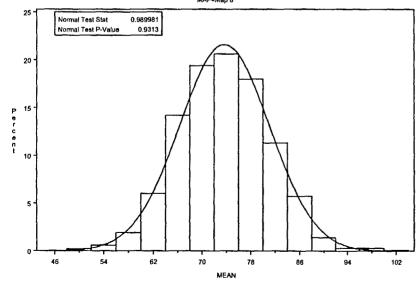


Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soll Grid Sampling Normality Fit, Arsenic, Mean of 30 Grabs MAP=Map 5



Normal Line: Mu=2375.8, Sigma=496.02

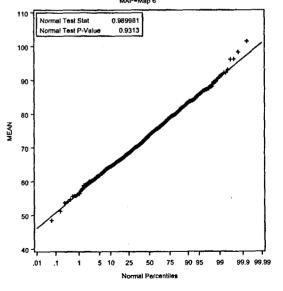
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 30 Grabs MAP=Map 6



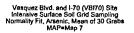
Curve:

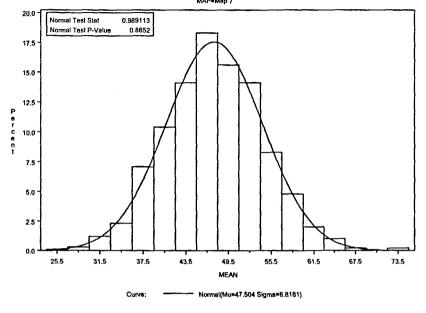
Normal(Mu=73.68 Sigma=7.3874)

Vasquez 8lvd. and I-70 (VBI70) Site Intensive Surface Scil Grid Sampling Normality Fit, Arsenic, Mean of 30 Grabs MAP=Map 6

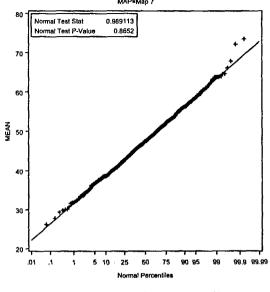


Normal Line: Mu=73.68, Sigma=7.3874



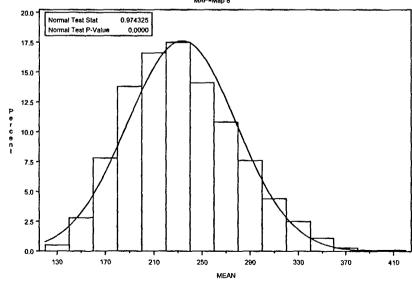


Vasquez Blvd. and I-70 (VBI70) Site intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 30 Grabs MAP=Map 7



Normal Line: Mu=47.504, Sigma=6.8181

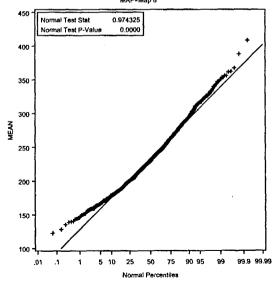
Vasquez Blvd. and 1-70 (VBI70) Site intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 30 Grabs MAP=Map 8



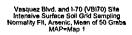
Normal(Mu=233.34 Sigma=45.258)

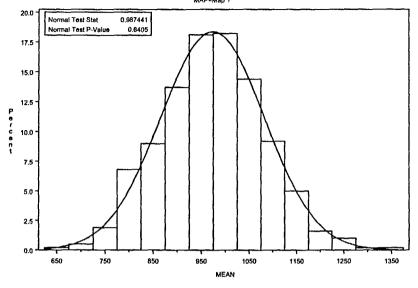
Curve:

Vasquez Bivd. and 1-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 30 Grabs MAP=Map 8



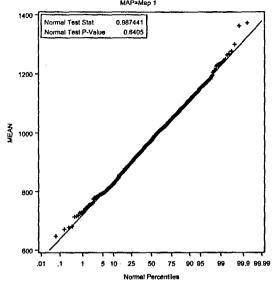
Normal Line: Mu=233.34, Sigma=45.258





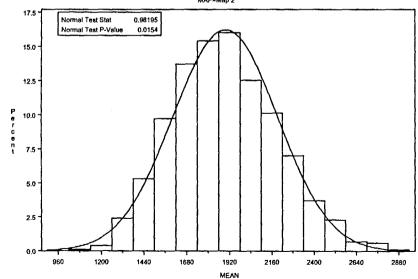
Curve: Normal(Mu=974,11 Sigma=108,68)

Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soll Grid Sampling Normality Fit, Arsenic, Mean of 50 Grabs MAP=Map 1



Normal Line: Mu=974.11, Sigma=108.68

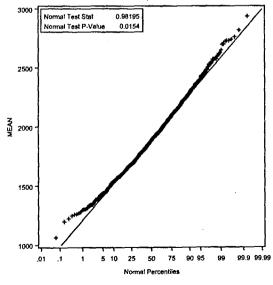
Vasquez Blvd. and 1-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsentic, Mean of 50 Grabs MAP≖Map 2



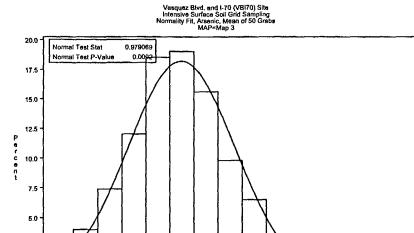
Normal(Mu=1897.7 Sigma=295.39)

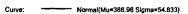
Curve:

Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 50 Grabs MAP=Map 2



Normal Line: Mu=1897.7, Sigma=295.39





MEAN

463

513

563

613

413

2.5

0.0

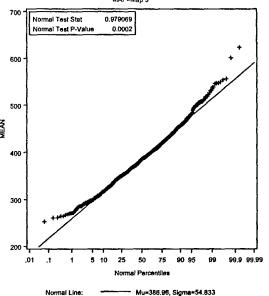
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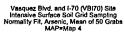
313

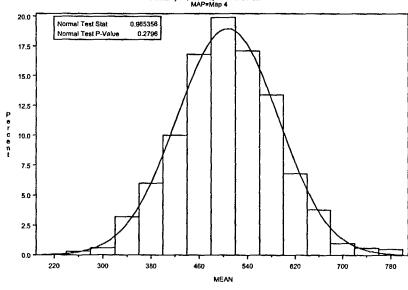
363

Curve:



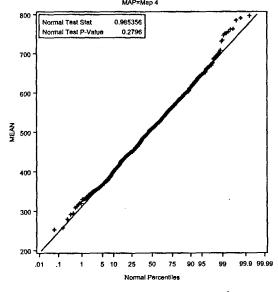




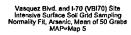


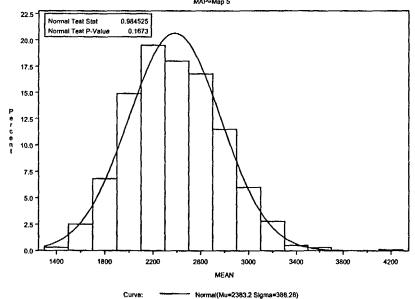
Normal(Mu=507.3 Sigma=84.123)

Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soll Grid Sampling Normalily Fit, Arsenic, Mean of 50 Grabs MAP≖Map 4

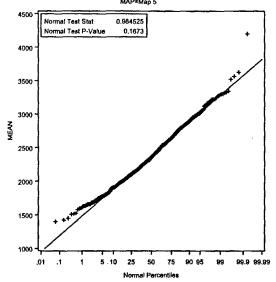


Normal Line: Mu=507.3, Sigma=84.123



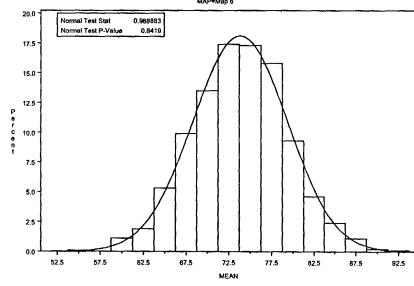


Vasquez BNd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 50 Grabs MAP≃Map 5



Normal Line: Mu=2383.2, Sigma=386.26

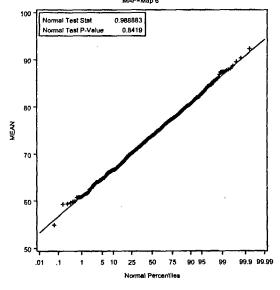
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 50 Grabs MAP≃Map 6



---- Normal(Mu=73.829 Sigma=5.5081)

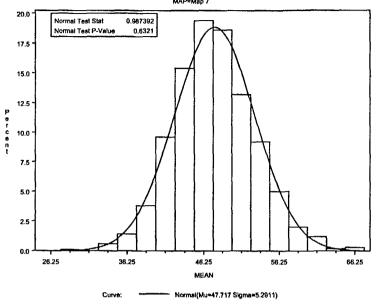
Curve:

Vasquez Bivd. and I-70 (VBI70) Site Intensive Surface Soll Grid Sampling Normality Fit, Arsenic, Mean of 50 Grabs MAP=Map 6

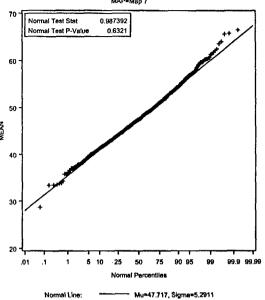


Normal Line: Mu=73.829, Sigma=5.5081

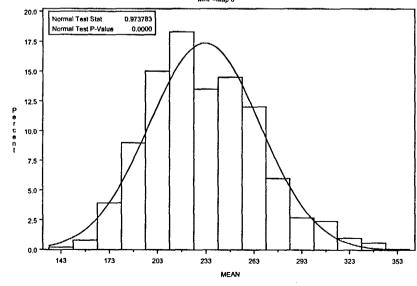




Vesquez Blvd. and 1-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 50 Grabs MAP=Map 7



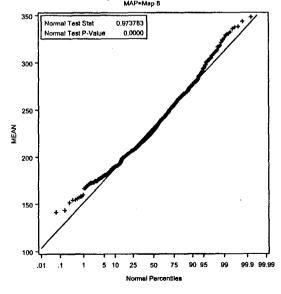
Vasquez Blvd. and 1-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 50 Grabs MAP=Map 8



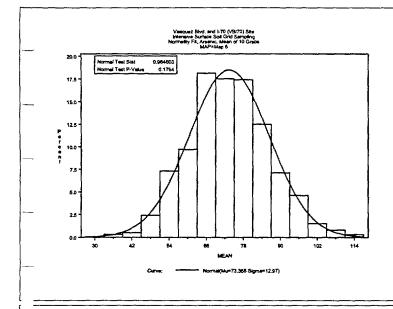
--- Normal(Mu=231.83 Sigma=34.468)

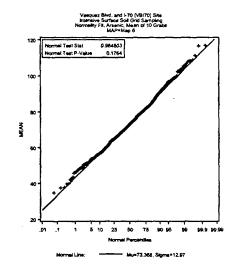
Curve:

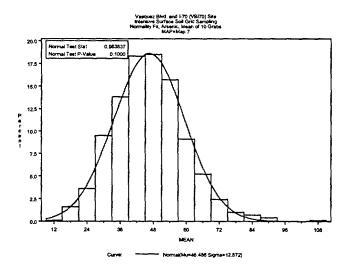
Vasquez Blvd. and I-70 (VBI70) Site Intensive Surface Soil Grid Sampling Normality Fit, Arsenic, Mean of 50 Grabs MAP=Map 8

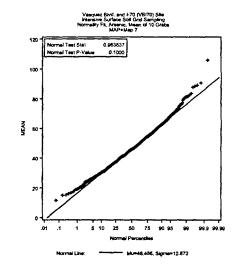


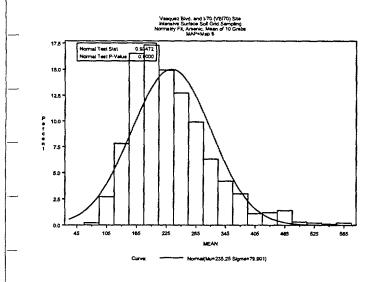
Normal Line: Mu=231.83, Sigma=34.468

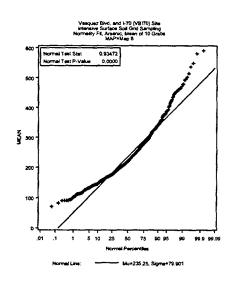


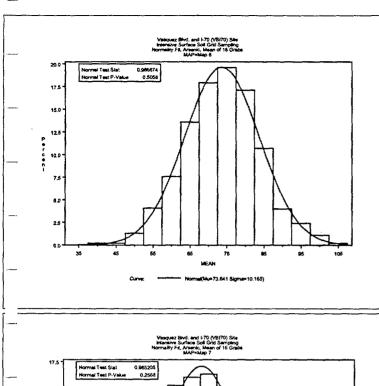


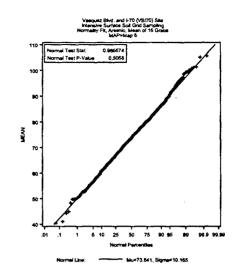


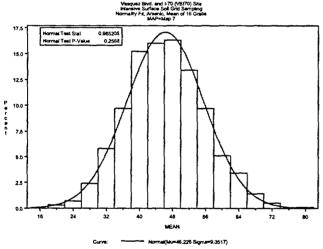


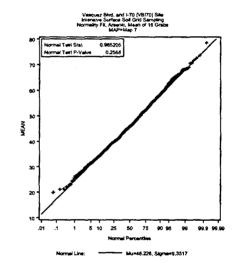


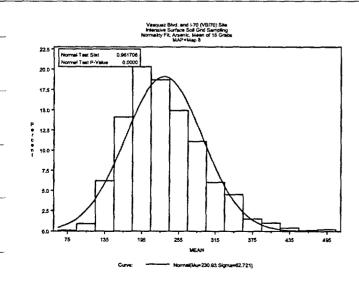


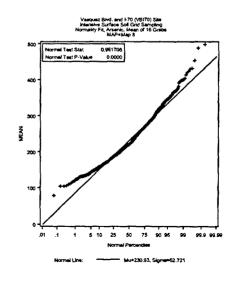


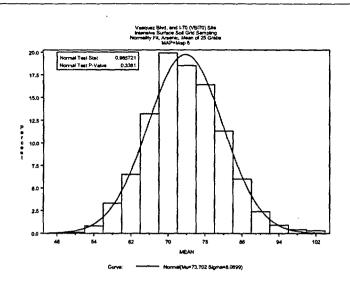


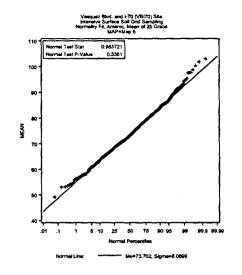


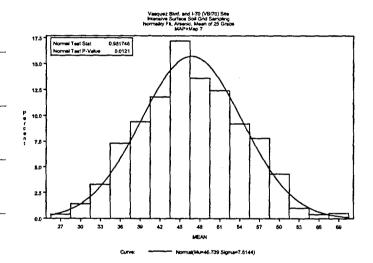


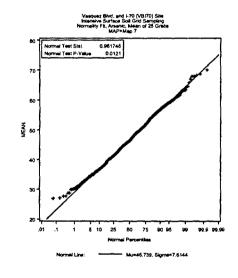


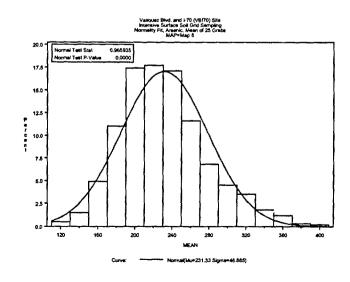


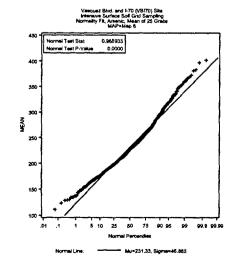


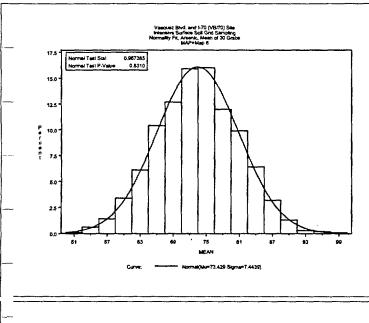


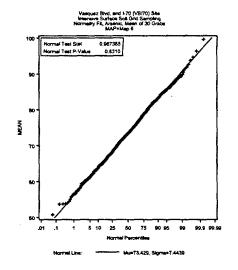


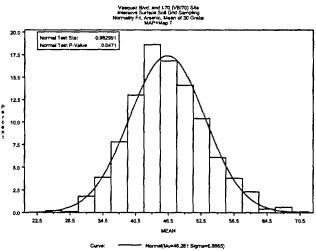


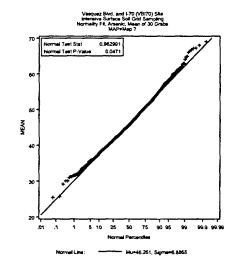


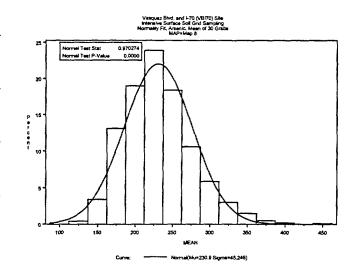


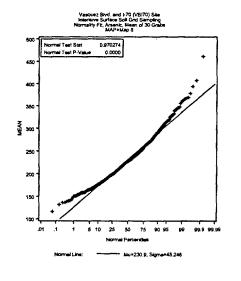


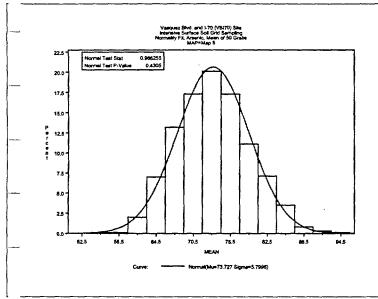


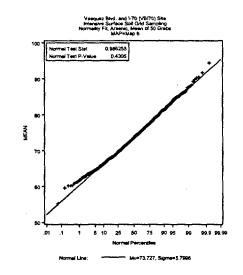


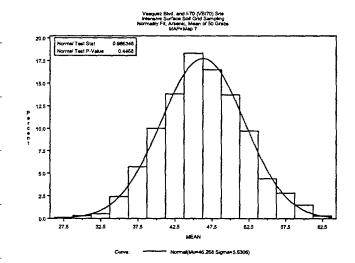


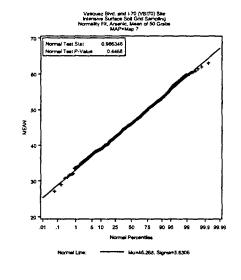


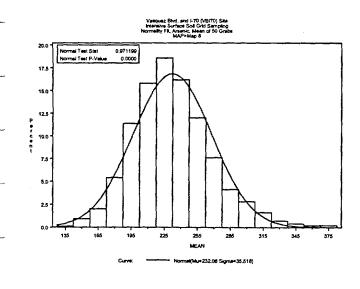


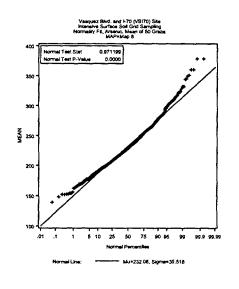












Appendix E:

Standard Operating Procedures

- Sample Identification and Tracking Procedures
- Residential Soil Sampling for Yard and Alleyway Soils
- High Volume Indoor Dust Sampling at Residences for Determination of Risk-Based Exposure to Metals
- Property Access
- Chain of Custody and Sample Handling
- Field Documentation
- Decontamination
- Investigation Derived Waste Management
- XRF Instrument Operation

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Title: SAMPLE IDENTIFICATION AND TRACKING PROCEDURES	
APPROVALS:	
Author ISSI Consulting Group, Inc. Ju	une 10, 19
	Date
III Surface Soil Investigation is provided.	for the Ph
III Surface Soil Investigation is provided. Received by QA Unit:	for the Pr
	for the Pr
Received by QA Unit:	for the Pr
Received by QA Unit: REVIEWS:	for the Pi

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SAMPLE IDENTIFICATION AND TRACKING PROCEDURES

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standardized method for uniquely identifying and tracking samples collected during the Phase III Surface Soil Investigation at the VBI70 site. This SOP is to be used by employees of USEPA Region 8 contractors/subcontractors supporting USEPA Region 8 projects and tasks. This SOP describes both the nomenclature which will be used to identify samples and outlines the measures by which samples will be tracked throughout the collection process. Site-specific deviations from the procedures outlined in this document must be approved by the EPA Region 8 Regional Project Manager or the Regional Toxicologist prior to initiation of the sampling activity.

2.0 RESPONSIBILITIES

Successful execution of the Project Plan requires a clear hierarchy of assigned roles with different sets of responsibilities associated with each role.

The Field Project Leader (FPL) may be an USEPA employee or contractor who is responsible for overseeing the sampling activities. The FPL is also responsible for checking all work performed and verifying that the work satisfies the specific tasks outlined by this SOP and the Project Plan. It is the responsibility of the FPL to communicate with the Field Personnel specific collection objectives and anticipate situations that require any deviation from the Project Plan. It is also the responsibility of the FPL to communicate the need for any deviations from the Project Plan with the appropriate USEPA Region 8 personnel (Regional Project Manager or Regional Toxicologist).

Field personnel performing sampling are responsible for adhering to the guidelines established within this SOP.

3. 0 SAMPLE NOMENCLATURE

All samples collected during this study will be assigned a unique label ("tag number").

Technical Standard Operating Procedures ISSI, Inc.
Contract No. SBAHQ-97-D-0003

SOP No. _____ Revision No.: 0 Date: 6/1999

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Each sample label will consist of three elements, as follows:

PHASE. All labels will begin with the number "3" to indicate that the sample is derived from Phase III of the study.

NUMBER. Each label will include a unique identification number. This number will be a 5-digit sequential number starting with "00001" and progressively increasing until the final sample has been collected or tag number "99999" has been reached.

SAMPLE PREPARATION. Samples will be categorized based upon the sample preparation performed. Categories include, but are not limited to:

- R Raw sample. Original sample collected during Phase III which is unprocessed.
- B Bulk fraction. The bulk soil fraction (sieved to < 2 mm).
- Fine fraction. The fine soil fraction (sieved to $< 250 \mu m$).

The sample preparation nomenclature may be expanded as needed in the future providing they are approved by the Project Database Manager or designate.

Thus, "3-00001-R" and "3-12846-F" represent possible sample numbers collected during Phase III.

4.0 SAMPLE TRACKING

Prior to sample collection, each team will be given blank copies of media-specific data sheets and a set of pre-printed sample identification numbers on self-adhesive labels. There will be two labels for each sample number. The set of labels that are checked out by a team will be documented by the FPL or designate prior to sampling each day.

When a sample of site medium is collected (e.g., yard soil, indoor dust, alleyway soil), a self-adhesive label will be transferred from the pre-printed sheet to the sample container. At the same time (before collection of any other sample), the second copy of the sample number will be transferred to the appropriate location on the data sheet. The sample data sheet will be filled out at the time of sample collection by the sample collection team. This sheet will contain all relevant information necessary to properly identify the sample.

Technical Standard Operating Procedures ISSI, Inc.
Contract No. SBAHQ-97-D-0003

SOP No. Revision No.: 0
Date: 6/1999

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An example data sheet is provided in Attachment 1. All data sheets will be maintained in three-ring binder logbooks. Each sampling team will have a separate logbook.

Because the sample identification number is not a self-reading or immediately decipherable, it is critical that the supporting sample data sheet be filled out legibly, accurately and completely. Notes should be as descriptive and as inclusive as possible such that a person reading the entries, who is independent of the sampling effort, should be able to reconstruct the sampling situation from the recorded information. Language should be objective, factual, and free of personal feelings and inappropriate terminology. Data sheets must be signed by the person recording the information. Any errors or mistakes in field recording must be initialed and dated by the recorder, along with a note explaining the change.

If self-adhesive labels are destroyed and/or voided during sampling activities, this information should be immediately documented in the general logbook for the field team.

5.0 DAILY CLOSE-OUT

Upon completion of daily sampling activities, the sampling team will return to the field office location with samples and corresponding data sheets and any unused labels. It is mandatory that each sample be submitted with its corresponding data sheet. The Field Project Leader or designated sample custodian will verify that the identification numbers on each sample correspond to the data sheet, and that each data sheet is legible and filled out in its entirety. Each data sheet will be copied and the originals will be transferred from the team logbook into a three-ring binder master logbook organized by sample identification number. Once inserted into the master logbook, each data sheet will be numbered sequentially in the space provided in the lower right corner. Additionally, the sample custodian will maintain a log of the sample identification numbers which have been used, noting any missing or destroyed labels (see Attachment 2). The sample labels and numbers for each team must be rectified at the end of each day. After verification, the samples will be locked and stored according to media. The copies of the sample data sheets will be submitted to the Field Database Manager for entry into the Field Activities Database. Data entry will be performed according to the Data Management Plan established for this project. A flowchart that illustrates the general flow of events is presented in Figure 1.

Technical Standard Operating Procedures ISSI, Inc.
Contract No. SBAHQ-97-D-0003

SOP No. _____ Revision No.: 0 Date: 6/1999

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Attachment 1:

VBI70 Surface Soil Sample Sheet

Technical Standard Operating Procedures ISSI, Inc.
Contract No. SBAHQ-97-D-0003

SOP No. ______ Revision No.: 0

Date: 6/1999

SURFACE SOIL DATA SHEET



PHASE:		3	_			
MEDIUM:	SUI	RFACE SOIL	_			
SOP:	ISSI-V	BI70-02 Revision 0	_			
DEPTH:		0-2"	-			
DATE:			-			
LOCATION	<u> </u>					
		House#	Street Name			
BUILDING 1	TYPE:	Residential	 Single Multifamily Apartment 			
		School	- Name			
		Park	- Name			
CLASS:		FS	-			
SAMPLE T	YPE:	COMP GRAB				
SAMPLE N	<u>o.:</u>					
Red						
Blue						
Yellow						
•	GARDE	EN PRESENT?	Yes I	No_		
	ADDRE	ESS CONFIRME	D BY RESIDENT?	Yes	No	
	WILLIN	IG TO ALLOW F	URTHER SAMPLING	i?	Yes	No
NOTES:						

Field Di	agram:		N †			
					. —	-

Samples Collected by:		
	Signature	Date
Logbook Page Reviewed by:		
	Signature	Date

sampleform: Page 2, 6/29/99

Page _____

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Attachment 2:

VBI70 Surface Soil Label Check-Out/Check-In Sheet

Technical Standard Operating Procedures ISSI, Inc.

Contract No. SBAHQ-97-D-0003

R:\Vasquez & I-70\Project Plans\SOPs\Phase III\Sample ID SOP\Sample ID SOP.doc

SOP No. ______ Revision No.: 0 Date: 6/1999

heck-Out/Check-In	Sheet
SOP-ISSI-VBI70-01 Revision 0	
3	1551
Soil	management of the second of th
0 - 2"	·
Start #	End #
	Date:
Start #	End #
	Date:
ed labels below. Be sure to	o document the Label # and
	SOP-ISSI-VBI70-01 Revision 0 3 Soil 0 - 2" Start #



VBI70 Surface Soil Labels - Master Sheet

	Check-Out		c	heck-In				
Label#	Sample Team ID	Date	Initials	Sample Team ID	Date	Initials	Voided?	Reason
3-00001								
3-00002								
3-00003						ļ		
3-00004								
3-00005		· · · · · · · · · · · · · · · · · · ·						
3-00006								
3-00007					ļ			
3-00008								
3-00009			ļ					
3-00010			ļ					
3-00011			ļ					
3-00012								
3-00013								
3-00014		-						
3-00015			<u> </u>				}	
3-00016								
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3-00018					·			
3-00019								
3-00020								
3-00021								
3-00022			ļ					
3-00023								
3-00024								7
3-00025								
3-00026								
3-00027								

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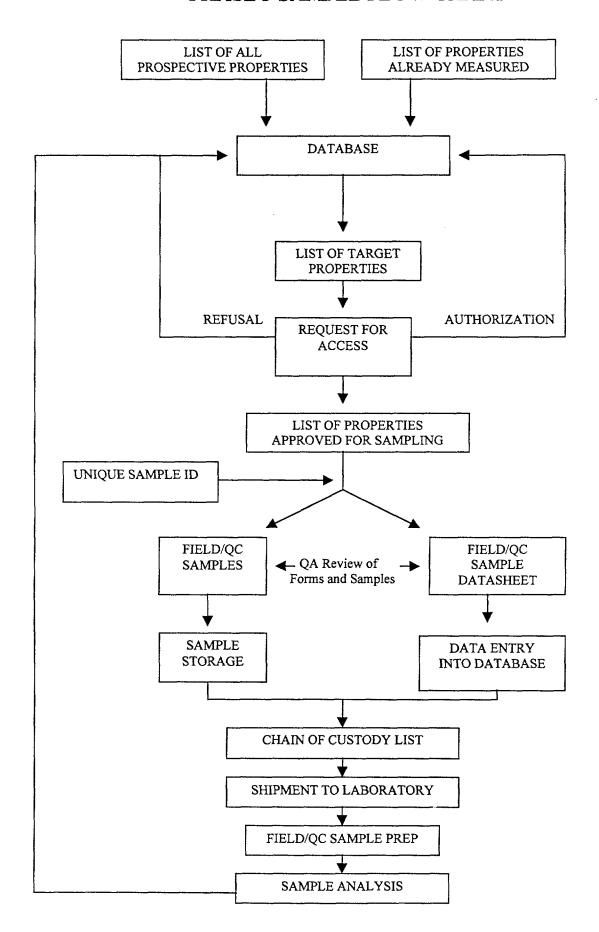
Figure 1:

Phase III Sample Flow Chart

Technical Standard Operating Procedures ISSI, Inc.
Contract No. SBAHQ-97-D-0003

SOP No. _____ Revision No.: 0 Date: 6/1999

PHASE 3 SAMPLE FLOW CHART



--DRAFT-

Date: June 14, 1999 (Rev. # 0)	S	OP No. <u>ISSI-VBI70-</u>
Title: RESIDENTIAL SOIL SAME PARK SOILS	PLING FOR YARD, ALLEYWA	AY, AND SCHOOL
APPROVALS:		
AuthorISSI Consulting	Group, Inc	June 14, 1999 Date
SYNOPSIS: A standardized method	d for exposure-based residential y	vard, alleyway, school
SYNOPSIS: A standardized method or park surface soil sampling compositing, and sample hand	is described. Protocols for samp	vard, alleyway, school le collection, sieving,
or park surface soil sampling	is described. Protocols for samp	vard, alleyway, school le collection, sieving,
or park surface soil sampling compositing, and sample han	is described. Protocols for samp	vard, alleyway, school le collection, sieving,
or park surface soil sampling compositing, and sample han Received by QA Unit:	is described. Protocols for samp	vard, alleyway, school le collection, sieving
or park surface soil sampling compositing, and sample han Received by QA Unit:	is described. Protocols for samp dling are provided.	le collection, sieving

Technical Standard Operating Procedures ISSI Consulting Group, Inc. Contract No. SBAHQ-98-D-0002

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SURFACE SOIL SAMPLING PROCEDURES

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standardized method for residential yard and alleyway surface soil sampling, to be used by employees of USEPA Region 8, or contractors and subcontractors supporting USEPA Region 8 projects and tasks. This SOP describes the equipment and operations used for sampling yard, alleyway and school or park surface soils in areas which will produce data that can be used to support risk evaluations. Deviations from the procedures outlined in this document must be approved by the USEPA Region 8 Regional Project Manager or Regional Toxicologist prior to initiation of the sampling activity.

2.0 RESPONSIBILITIES

Successful execution of the Project Plan requires a clear hierarchy of assigned roles with different sets of responsibilities associated with each role.

The Field Project Leader (FPL) may be an USEPA employee or contractor who is responsible for overseeing the residential yard and alleyway surface soil sampling activities. The FPL is also responsible for checking all work performed and verifying that the work satisfies the specific tasks outlined by this SOP and the Project Plan. It is the responsibility of the FPL to communicate with the Field Personnel regarding specific collection objectives and anticipated situations that require any deviation from the Project Plan. It is also the responsibility of the FPL to communicate the need for any deviations from the Project Plan with the appropriate USEPA Region 8 personnel (Regional Project Manager or Regional Toxicologist).

Field personnel performing residential yard, alleyway and school or park soil sampling are responsible for adhering to the applicable tasks outlined in this procedure while collecting samples. The field personnel should have limited discretion with regard to collection procedures, but should exercise judgment regarding the exact location of the Sample Point, within the boundaries outlined by the FPL.

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3.0 EQUIPMENT

- Soil coring tool Various models of coring tools are acceptable and selection of the specific brand and make of tool should be specified in the Project Plan. Selection of the coring tool should be based on the individual characteristics of the soil to be sampled (e.g. clay, stony, soft etc.). At a minimum, the tool should be capable of retrieving a cylindrical plug of soil 3 inches in diameter and 3 inches long. A soil coring tool of this type is typically fabricated from stainless steel, has a hollow stem, is T-shaped and uses two handles to apply the force necessary for core collection. A plunger is used to press out the soil plug from the tip of the coring device. Plungers may be fitted with an adjustable stop to allow all but a given length of soil to be pushed from the coring tool. In all cases the procedures recommended by the manufacturers should be followed with regard to use of the coring tool. Coring tools with disposable plastic sleeves may be employed to minimize the decontamination effort.
- <u>Collection containers</u> plastic zip-lock bags. Containers may also be glass jars or plastic jars.
- <u>Scoop/spoon</u> for collecting surface soil samples. May be plastic or stainless steel. Must be lead free and unpainted.
- <u>Gloves</u> for personal protection and to prevent cross-contamination of samples. May be plastic or latex. Disposable, powderless.
- <u>Field clothing and Personal Protective Equipment</u> as specified in the Health and Safety Plan.
- <u>Squeeze bottle</u> -for dispensing potable (drinking) quality water. Used to clean and decontaminate sampling equipment.
- <u>Squeeze bottle</u> for dispensing deionized water. Used to clean and decontaminate sampling equipment.
- <u>Sampling Flags</u> red, blue, and yellow. Used for identifying yard soil sampling locations. Each color represents a different composite sample.
- <u>Wipes</u> disposable, paper or baby wipes. Used to clean and decontaminate sampling equipment and flags.
- <u>Field notebook</u> -a bound book used to record progress of sampling effort and record any problems and field observations during sampling.

Technical Standard Operating Procedures ISSI Consulting Group, Inc. Contract No. SBAHQ-98-D-0002

SOP No. <u>ISSI-VBI70-02</u> Revision No.: 0 Date: 6/1999

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- <u>Field Data Collection Sheet</u> a three-ring binder book of forms used to record and track samples collected at the VBI70 site. An example form is provided in Attachment 1.
- <u>Permanent marking pen</u> used to label sample containers.
- Sieves U.S. Standard # 10 (capable of passing material < 2 mm) and U.S. Standard # 60 (capable of passing material < 250 μ m). Used to remove gravel and debris in the field to minimize shipping weight. Sieves mesh should be constructed of stainless steel or plastic and designed for soil processing.
- Measuring tape or pocket ruler used to measure the length of soil core in the soil coring device.
- <u>Plastic Buckets</u> used to receive rinse water generated in the course of tool cleaning, rinsing sieves, and used to collect the discarded soil from the coring tool. One bucket should be set aside to store marker flags.
- <u>Trash Bag</u> used to dispose gloves and wipes.
- <u>0.01M HCl</u> used for equipment decontamination.

4.0 SAMPLING PATTERN

Sampling patterns for residential yard, alleyway, school or park soils are designed to identify and collect samples to support human health risk assessment. Idealized sampling patterns for residential and alley soils are presented in the attached figures, but possible deviations from these sampling patterns could occur based on site-related issues such as additional buildings on a property (e.g., garage), the shape of the property, or the shape of the house. If deviations from the idealized sampling pattern occur, they should be noted in the Sample ID logbook along with a reason for deviating from the original sampling pattern. Proposed sampling patterns for the individual schools and parks will be provided as an attachment to the Phase III Field Investigation Project Plan.

4.1 RESIDENTIAL YARD SOIL

Composite sampling requires soil collection from multiple (sub-sample) points. These soils are then mixed and used as a measure of the concentration averaged over the entire area (zone). Surficial yard soil samples (0-2 inches) will be collected.

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Soil Sample Location and Compositing

The surficial sampling locations within a yard will be based on a 30-point sampling grid. Because of the large number of properties that require sampling during this project, an independent chemical analysis will not be performed for each of the sub-samples collected from each property. Rather, three composite samples will be collected per residence, each consisting 10 sub-samples that are identified by randomly selected flags (e.g., 10 red, 10 blue, and 10 yellow). Identification of individual grab sample locations will be performed using the standardized method as follows.

The FPL or designate will be trained in this procedure in order to ensure replicable sample location assignment. There are four major steps in grab sample location identification. They are:

- Draw a field diagram of the property and its major components approximately to scale
- Estimate the samplable area in the yard
- Divide the samplable yard into 5 subsections
- Place 6 flags in each subsection

Field Diagram

The FPL or designate will visit a residence at the time of sampling to assign the sampling scheme. The FPL will pace off the major attributes of the residence (e.g., dimensions of the property boundary, house, garage, driveway, etc.) and prepare a field diagram to approximate scale (± 5 feet on each measurement). The goal is not have a drawing to scale, but instead to have an estimate of the total samplable area in the residential yard. Figure 1 provides an example of a typical residence at the VBI70 site that has been drawn on a grid.

Estimate the Area in the Yard

The total area of the yard that is available for sampling will be estimated by counting the number of visible squares (grids) that comprise the yard (e.g., squares that are not taken up by the house, garage and other obstructions). The total number of squares will be estimated to the ½ square. In the example (Figure 1), the samplable area is approximately 45 squares.

Divide the Yard Area into Subsections

The samplable property will be divided into 5 subsections of approximately equal area using natural boundaries such as the house, garage, sidewalk or gardens as division markers (See Figure 2). In the example, each subsection is made up of about 9 squares (\pm 1 square).

Technical Standard Operating Procedures ISSI Consulting Group, Inc. Contract No. SBAHQ-98-D-0002

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Flag Placement in Each Subsection

As discussed previously, a total of 30 samples will be identified using marker flags of any three different colors (e.g., 10 red, 10 blue and 10 yellow). Six (6) marker flags (2 red, 2 blue and 2 yellow) will be used to identify each sample location within a subsection. As seen in Figure 2, the locations of each marker flag should be approximately equidistant from the other flags within each subsection. Additionally, each color flag should be alternately placed so that same color marker flags are not clustered.

Yard Soil Sampling

The first 10-point composite will be collected by combining the samples at flags of similar color (e.g., red). Grab samples will be collected from the 0-2 inch soil horizon approximately 3-6 inches from each marker flag. Each sample will be collected using a clean coring tool (3-inch diameter). Each grab sample marked by a red flag will be placed into a single ziplock bag and labeled in accord with the Sample Identification and Tracking SOP# ISSI-VBI70-01 Revision 0. Because property sizes and obstacles present at each residence may vary significantly, actual sample locations will be identified using a diagram that will be drawn for each individual property sampled. If obstructions are present at the intended sampling locations (e.g., sidewalk, shed, garden, etc.), the sample point should be offset so that a surficial yard soil may be collected, then the actual sample location must be correctly documented on the field diagram.

Equipment decontamination (decon) must occur between collection of each composite if clean equipment tools are not available. Decon procedures are described in Section 10.0. Once sample collection at the red marker flags is complete, all subsamples taken from the 10 locations identified with a blue flag will be collected and placed in a single ziplock bag and labeled appropriately. Decontamination will be performed as appropriate. Finally, all subsamples taken from the 10 locations identified with a yellow flag will be placed in a single ziplock bag and labeled appropriately. Decontamination will be performed as appropriate.

Care should be taken to avoid tracking soil from one area to another. As samples are taken sequentially, care should also be taken not to contaminate an area yet to be sampled with the residue of the sample that is currently being taken. In general, one should move in a single direction through the sampling area. If an area is known or suspected of having a higher concentration of metals, all other considerations being equal, it should be sampled last to prevent cross contamination.

4.2 ALLEYWAY SOIL

Currently, the relationship between a residence with elevated (>200 ppm) arsenic concentrations in yard soil and possible adjacent alleyways is not understood. For the purposes of this pilot investigation, a minimum of four and a maximum of six alleyway units located adjacent to a

Technical Standard Operating Procedures ISSI Consulting Group, Inc. Contract No. SBAHQ-98-D-0002

SOP No. <u>ISSI-VB170-02</u> Revision No.: 0 Date: 6/1999

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residence with elevated (>200 ppm) arsenic concentration will be identified and sampled. Priority will be given to locations where a composite has been collected from all or most of the properties in the study location, and where indoor dust samples have also been collected. Grab samples will be collected in a pattern similar to that shown in Figure 3. The initial sampling location (alleyways) will be recorded and documented on the sample data sheet field diagram.

Prior to sampling the FQAC or designate will provide maps that identify the alleyways and individual sample locations using GIS tools. The map will be used to identify and document sample locations. In the event that sample locations must be offset due to presence of obstructions, the new location must be marked on the map. Grab sample locations will be placed along a center transect of each residential property along the alleyway, three samples will be collected across the alley. Approximately thirty grab samples for the entire block (see Figure 3). The three samples are located in the center and two sides of the alley where the two sides are about 2 feet from the property line of residences that border the alleyway. Grab samples will be collected from the 0-2 inch soil horizon approximately 3-6 inches from the each marker flag. Each sample will be collected using a clean coring tool (3-inch diameter) and placed into a separate ziplock bag. The samples will be identified as a unique number (e.g., 1 through 30). Each sample number must be placed in the appropriate box on the alleyway soil sample data sheet, as well as on the zip-lock bag. Sample labeling procedure is described in SOP ISSI-VBI70-01 Revision 0.

Care should be taken to avoid tracking soil from one area to another. As samples are taken sequentially, care should also be taken not to contaminate an area yet to be sampled with the residue of the sample that is currently being taken. In general one should move in a single direction through the sampling area. If an area is known or suspected of having a higher concentration of metals, all other considerations being equal, it should be sampled last to prevent cross contamination.

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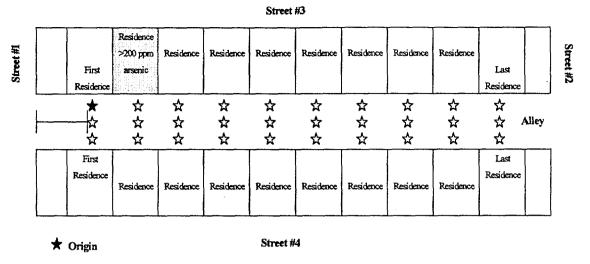


Figure 3 Idealized Alleyway Sampling Strategy

4.3 SCHOOLS AND PARKS SOIL

Surface soil samples at schools and parks will be collected using the same sampling strategy as discussed for the residential soil sampling (Section 4.1). The number of grab samples collected at an individual school or park may vary, but 3 composite samples will be collected at minimum. Each individual grab sample will be identified using marker flags of any three different colors (e.g., red, blue and yellow). The exact sampling pattern will be unique to the individual school or park, and will be submitted as an attachment to the Project Plan at a later date. At minimum, each marker flag will be approximately equidistant from the other flags and each color flag should be alternately placed so that same color marker flags are not clustered.

The first composite will be collected by combining the samples at flags of similar color (e.g., red). Grab samples will be collected from the 0-2 inch soil horizon approximately 3-6 inches from each marker flag. Each sample will be collected using a clean coring tool (3-inch diameter). Each grab sample marked by a red flag will be placed into a single ziplock bag and labeled in accord with the Sample Identification and Tracking SOP# ISSI-VBI70-01 Revision 0. If obstructions are present at the intended sampling locations (e.g., sidewalk, garden, etc.), the sample point should be offset so that a surficial soil may be collected, then the actual sample location must be correctly documented on the field diagram.

Equipment decontamination (decon) must occur between collection of each composite if clean equipment tools are not available. Decon procedures are described in Section 10.0. Once sample collection at the red marker flags is complete, all subsamples taken from the 10 locations identified with a blue flag will be collected and placed in a single ziplock bag and labeled appropriately. Decontamination will be performed as appropriate. Finally, all subsamples taken from the 10 locations identified with a yellow flag will be placed in a single ziplock bag and labeled appropriately. Decontamination will be performed as appropriate.

Technical Standard Operating Procedures ISSI Consulting Group, Inc. Contract No. SBAHQ-98-D-0002

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Care should be taken to avoid tracking soil from one area to another. As samples are taken sequentially, care should also be taken not to contaminate an area yet to be sampled with the residue of the sample that is currently being taken. In general, one should move in a single direction through the sampling area. If an area is known or suspected of having a higher concentration of metals, all other considerations being equal, it should be sampled last to prevent cross contamination.

5.0 COLLECTION OF COMPOSITE SAMPLES USING A CORING TOOL

A new pair of plastic gloves are to be worn in each Sampling Zone. For example, a new pair of gloves should be worn for each composite sample at each residential yard.

Locate the Sub-sample Point on the ground specified by the Project Plan and clean the area free of twigs, leaves, and other vegetative material that can be easily be removed by hand. If the specified Sub-sample Point is occupied by a rock, cobble or other hard object of sufficient size to be incapable of easy removal by hand, move the Sub-sample Point to a location closest to the original Sample Point and document the change in sample location in the field logbook page.

Place the soil coring tool on the ground and position it vertically. Holding the tool handle with both hands, apply pressure sufficient to drive the tool approximately three inches into the ground while applying a slight twisting force to the coring tool. Remove the tool by pulling up on the handle while simultaneously applying a twisting force. If the sample was retrieved successfully, a plug of soil approximately three inches long should have been removed with the coring tool.

If the Project Plan calls for coring of soil covered by turf-like vegetation (lawn), the coring tool should be pushed through the sod and the root mass extracted along with the soil core.

Hold the soil coring tool horizontally or place it on the ground. Place the coring tool plunger with the two inch stop inside the coring tool and push the soil plug out of the coring tool until the stop is encountered and two inches of soil remains inside. Using a clean spatula or knife, remove the soil collected at depth greater than two inches from the end of the sampling tool. Allow this soil to fall into the plastic bucket designated for excess soil material. Remove the stoppered plunger from the soil coring tool and using the unstoppered plunger, push the two-inch soil plug from the coring tool so that it falls directly into the sample container. Repeat the steps outlined above until all the sub-samples from a given zone have been collected in the sample container.

Decontaminate equipment as described in Section 10.0.

6.0 COLLECTION OF COMPOSITE SAMPLES USING A SPOON OR SCOOP

A new pair of plastic gloves are to be worn in each Sampling Zone.

Locate the Sub-sample Point on the ground specified by the Project Plan and clean the area free of twigs, leaves, grass, and other vegetative material that can be easily be removed by hand. If

Technical Standard Operating Procedures ISSI Consulting Group, Inc. Contract No. SBAHQ-98-D-0002

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the specified Sub-sample Point is occupied by a rock, cobble or other hard object of sufficient size to be incapable of easy removal by hand, move the Sub-sample Point to a location closest to the original Sample Point.

Using the metal spoon or scoop, excavate a hole in the soil approximately two inches in diameter and two inches deep while placing the excavated material directly inside the compositing bowl. The sides of the excavated hole should be close to vertical to avoid sampling that is biased in favor of the upper layer of soil.

Repeat steps outlined above until all the sub-samples from a given zone have been collected in the sample container.

Decontaminate equipment as described in Section 10.0.

7.0 SITE CLEAN-UP

The Project Plan will address the methods used to fill holes generated by the sampling procedure. In general, it is desirable to fill sampling holes with clean, moist topsoil. The material should be poured into the hole and tamped down lightly.

Rinse water and 0.01M HCl, the unused fraction of soil cores, the roots of vegetation removed during sampling, and any unused soil generated in the course of sieving must be disposed of as specified in the Project Plan. Unless otherwise determined, this material should be regarded as hazardous waste and disposed accordingly.

All 30 flags (if reused) should be decontaminated by wiping off with towels and/or baby wipes.

8.0 RECORDING KEEPING AND QUALITY CONTROL

A general field notebook should be maintained by each team that is collecting samples as described in the Project Plan. Additionally, each team will maintain a sample ID logbook. The following information should be collected and maintained in each:

General Field Logbook

- date
- time
- personnel/team members
- weather conditions
- descriptions of any deviations to the Project Plan and the reason for the deviation.

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Sample ID Logbook

- a sketch of the sampling pattern for each residence
- Sample ID numbers
- QC samples collected
- description of any deviations and reasoning why

Field drawings with grid dimensions needed for 30 subsamples size should integrate the approximate house location and size. The diagram should also depict the 30 subsamples (by color, sample location, and sample number). In addition, samples taken from soils with visible staining or other indications of non-homogeneous conditions should be noted.

Field personnel will collect the proper type and quantity of quality control samples as prescribed in the Project Plan.

9.0 SAMPLE PREPARATION

Because data generated from collected surface soils will be used in evaluations of risk for metals exposure, sieving is required to obtain particle sizes that are the primary source of human exposure ($<250~\mu m$). However, due to the large number of samples planned for the Phase III sampling event, only a portion of samples will be sieved to $<250~\mu m$. The frequency of samples identified for fines is outlined in the Project Plan. Soil sample must be dried and sieved in a controlled environment (laboratory) rather than in the field. Composite samples should have their sub-samples mixed prior to sieving.

9.1 Drying the Soils

Soils must be sufficiently dry prior to sieving. This may be determined by performing a "squeeze" test. The soil plug is pinched between a freshly gloved thumb and index finger. If the soil fragments and becomes powdery, the sample may be regarded as adequately dry for sieving. Alternatively, if soil squeezed in the palm of a freshly gloved hand becomes cohesive and retains its shape after squeezing, the soil has too much moisture for sieving.

If samples are not sufficiently dry, they should be air-dried by being allowed to stand in an open or partially covered sample container for 24 hours. Air-drying should be carried out in a warm room with moderate air circulation. If the soil is still too moist, it should be left to air dry for another 24 hours and tested again.

Rough guidelines for soil drying times are as follows:

- Sandy soil (24 hours)
- Silty soil (24 48 hours)
- Clayey soil (36 60 hours)

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Once soil samples have been determined to be adequately dry, the sample plug or scoop should be manually crushed and broken up by squeezing the material with a freshly gloved hand. If the sample contains a section of grass sod, the soil should be shaken from the grass roots allowing this soil to mix with the other soil that will be sieved. The grass sod plug should be subjected to the screening process along with the other soil. Under no circumstances should the sample be ground (either against itself or against the compositing bowl or the sieving screens) as grinding generates particles that would not otherwise exist as part of the soil matrix.

9.2 Sieving

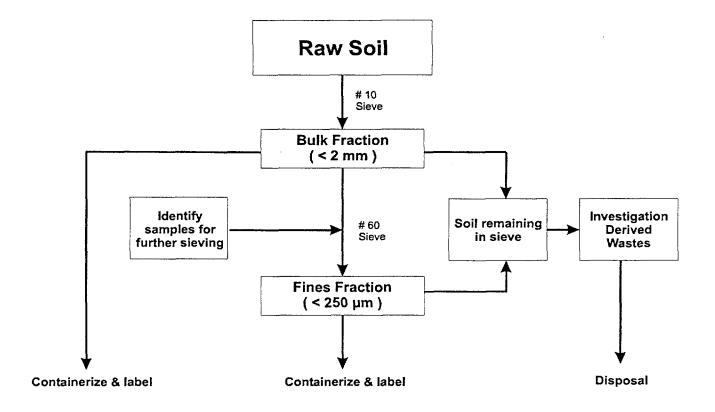
Sieving will be performed for each sample using clean equipment as outlined in Figure 4. Unprocessed soils (defined here as "raw soil") should first be sieved using a #10 screen, allowing particles <2 mm to pass through its mesh. Soils passing through a #10 screen will be defined here as "bulk soil". Upon request, the bulk soil should then be sieved using a #60 screen, allowing particles $<250 \mu m$ to pass through its mesh. Soils passing through a #60 screen are referred here as fine soil ("fines"). The screens may be stacked with the #10 screen on top and the #60 screen below. Covers (top and bottom) may be used as part of the sieving process if they are designed as part of the sieve set.

Sieving should be performed by pouring the soil sample on top of the sieve and shaking the screen rapidly back and fourth so that the material rolls over the screen mesh. The screen should occasionally be tapped against a hard surface to allow material to pass through mesh holes that have become clogged. Shaking should continue only as long as material above the screen contains particles smaller than the mesh opening. The screening process should not be used to break-up fragments of the soil core and materials should not be rubbed against the screen as a way of making them pass through the mesh.

The screens should be thoroughly cleaned prior each use. Decontamination procedures are described in Section 10.0.

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Figure 4: Soil Preparation Flow Chart



10.0 DECONTAMINATION

Because decontamination procedures are time consuming, having a quantity of sampling tools sufficient to support decontamination at a maximum of once per day is recommended. All sampling and sieving equipment must be decontaminated prior to reuse.

The procedure to decontaminate all equipment is outlined below:

- 1) Remove visible soil.
- 2) Rinse equipment with potable water.
- 3) Rinse equipment with deionized water.
- 4) Rinse in a solution of 0.01M HCl.
- 5) Final rinse with deionized water.

Washing should be performed by sequential immersion of the equipment in buckets partially filled with these solutions. If necessary, a brush should be used to remove soil material from screens and coring tools. Equipment should be set on clean toweling to dry. Equipment should be visibly dry before being used again.

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Wipes, gloves, and rinse solutions must be disposed or stored properly as specified in the Project Plan.

11.0 GLOSSARY

- <u>Project Plan</u> A written document that spells out the detailed site-specific procedures to be followed by the FPL and the field personnel.
- Sample Point The actual location at which the sample is taken. The dimensions of a sample Point are 3" in diameter and 2" deep (core technique) or 3" across by 2" deep (spoon/scoop technique).
- <u>Composite Sampling</u> A sample program in which multiple Sample Points are compiled together and submitted for analysis as a single sample.
- <u>Sample zone</u> A unit of surface area subjected to a given sample program. A given zone usually is thought to contain similar metals concentrations or to be defined by a single set of exposure parameters.
- Raw soils Soil with sticks, leaves and debris removed but otherwise unprocessed.
- <u>Bulk soils</u> Raw soil that has passed through a U.S. Standard #10 sieve (< 2 mm).
- Fine soil Bulk soil that has passed through a U.S. Standard #60 sieve ($< 250 \mu m$).

12.0 REFERENCES

USEPA, 1995. Residential Sampling for Lead: Protocols for Dust and Soil Sampling, Final Report, EPA 747-R-95-001, USEPA, March 1995, 38 p.

American Society for Testing and Materials, 1995. Standard Practice for Field Collection of Soil Samples for Lead Determination by Atomic Spectrometery Techniques, ASTM Designation: E 1727 - 95, October 1995, 3 p.

Figure 1 Proposed Grid Sampling Design for Residential Surface Soil

Step 1:

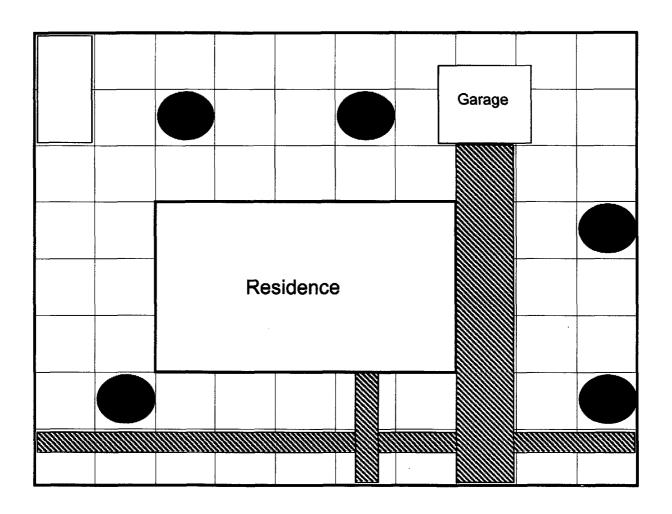
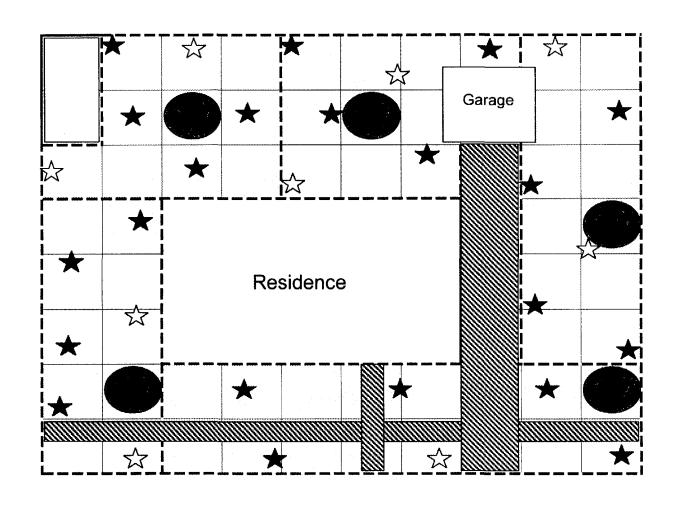
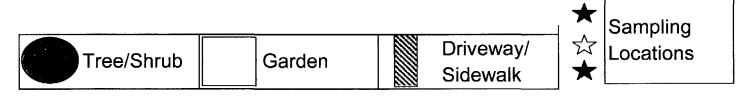




Figure 2 Proposed Grid Sampling Design for Residential Surface Soil Step 2:





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Attachment 1

Technical Standard Operating Procedures ISSI Consulting Group, Inc. Contract No. SBAHQ-98-D-0002

SOP No. <u>ISSI-VBI70-02</u> Revision No.: 0 Date: 6/1999

SURFACE SOIL DATA SHEET



PHASE:	3			
MEDIUM:	SURFACE SOIL			
SOP:	ISSI-VBI70-02 Revision 0			
DEPTH:	0-2"	•		
DATE:				
LOCATION:			_	
	House#	Street Name		
BUILDING 1		 Single Multifamily Apartment 		
	School	- Name	-	
	Park	Name	-	
CLASS:	FS			
SAMPLE TY	/PE: COMP GRAB			
SAMPLE NO	D.:			
Red				
Blue				
Yellow				
	GARDEN PRESENT? ADDRESS CONFIRMEI WILLING TO ALLOW F		Yes No Yes	No No
NOTES:		 		

Field Diagram:		N †			
				:	

Signature

Signature

sampleform: Page 2, 6/29/99

Samples Collected by:

Logbook Page Reviewed by:

Page _____

Date

Date

DON		
DCN		

VBI70 Alleyway SOIL DATA SHEET



PHASE: 3	MEDIUM:	Alley Soil
DATE:	DEPTH:	0-2"
LOCATION:	SOP:	ISSI-VBI70-02 Revision 0
LENGTH OF ALLEY: INTER-SAMPLE DISTANCE:		

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Date: <u>June 28, 1999 (Rev. # 0)</u>	S	OP No. <u>ISSI-VBI70-03</u>
Title: HIGH VOLUME INDOOR DETERMINATION OF RIS		
APPROVALS:		
Author ISSI Consulti	ng Group, Inc.	June 28, 1999 Date
SYNOPSIS: A standardized high-residences is described. This concentration (mg/kg) or con-	s method is suitable for m	easurement of either contaminant
Received by QA Unit:		
REVIEWS:		
TEAM MEMBER	SIGNATURE/TITLE	DATE
USEPA Region 8		
ISSI Consulting Group, Inc.		

Technical Standard Operating Procedures ISSI Consulting Group, Inc. Contract No. SBAHQ-98-D-0002

SOP No. VBI70-03 Revision No.: 0 Date: 6/1999

INTERIOR SURFACE DUST SAMPLING AT RESIDENCES

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to provide a standard approach for collection of interior surface dust samples within a residence. The SOP includes a description of the equipment and methods to be used. This protocol will be implemented by employees of USEPA Region 8 or contractors and subcontractors supporting Region 8 projects and tasks.

2.0 RESPONSIBILITIES

The Field Project Leader (FPL) is responsible for ensuring that all dust samples collected are obtained in accord with the procedures specified in this SOP. The FPL may be an USEPA employee or an USEPA contractor. The FPL is responsible for training all Field Personnel in the methods and techniques specified in this SOP and for checking that all work performed satisfies the specific tasks outlined by this SOP and the Project Plan. It is the responsibility of the FPL to identify any deviations from the SOP that may be required and to obtain approval for these deviations from the USEPA Region 8 Remedial Project Manager, Regional Toxicologist, or Field Quality Assurance Coordinator prior to initiation of any sampling activities that are not in accord with this SOP.

3.0 DUST COLLECTION PROTOCOL

3.1 Overview

This protocol is for collection of dust samples from interior surfaces using a high-volume vacuum method. The sampling method is based on the method of Roberts et al. (1989, 1991, 1994) and Stamper et al. (1990), and is presented in ASTM's Standard Practice for Collection of Dust from Carpeted Floors for Chemical Analysis (ASTM 1993). The protocol is suitable for the collection of interior dust samples from either hard or smooth and highly textured surfaces, such as brickwork and rough concrete, and soft, fibrous surfaces, such as upholstery and carpeting.

At the VBI70 site, one dust sample will be collected at each residence. This sample will be a composite of dust collected from multiple different sub-locations within the residence. At each sub-sampling location, dust is withdrawn from the surface area by means of a flowing air stream passing through a sampling nozzle at a specific velocity and flow rate. Dust is separated from the air mechanically by a cyclone and is collected in a catch bottle attached to the bottom of the cyclone. The cyclone collects particles

approximately 5-µm mean aerodynamic diameter and larger. The collected sample is substantially unmodified by the sampling procedure.

3.2 SAMPLING EQUIPMENT

3.2.1 Sampling Apparatus

The sampling apparatus may be acquired commercially [CS₃ model HVS3] (see Figure 1) or constructed. The dimensions of the sampling apparatus (nozzle size, cyclone diameter, cyclone inlet diameter, etc.) are interdependent. The flow rate must produce a sufficient velocity both at the sampling surface and in the cyclone. The cyclone must have a cut diameter of 5 µm at the same velocity that will provide a horizontal velocity of 40 cm/s at 10 mm from the nozzle in the carpet material. The fundamental principles of this device have been discussed in Roberts et al. (ASTM 1994).

- Nozzle The edges and corners of the sampling nozzle shall be rounded to prevent catching any carpeted material. The nozzle must be constructed to allow for sufficient suction to separate loose particles from the carpet and carry them to the cyclone. It must have an adjustment mechanism to establish the nozzle lip parallel to the surface and to achieve the proper suction velocity and pressure drop across the nozzle. A nozzle 12.4 cm long and 1 cm wide, with a 13-mm flange and tapered to the nozzle tubing at no more than 30°, will yield the appropriate velocities when operated as specified.
- Gaskets Gaskets in joints should be of a material appropriate to avoid sample contamination.
- Cyclone The cyclone shall must be constructed such that air flow allows for separation of particles 5-µm mean aerodynamic diameter and larger. The cyclone must be made of
- aluminum or stainless steel.
- Catch Bottle The catch bottle must be either a 500-mL amber glass jar (Fisher Scientific Cat. No. 03-320-4C) or 500-mL fluorinated ethylene propylene (FEP) bottle (Fisher Scientific Cat. No. 03-312-22) to avoid contamination and allow the operator to see the sample.
- Flow Control System The flow control system shall allow for substantial volume adjustment. The suction source must be capable of drawing 12 L/s through the system with no restrictions other than the nozzle, cyclone, and flow control system connected. A commercial vacuum cleaner can be used for this purpose.
- Flow Measuring and Suction Gages The use of Magnehelic gages for measurement of the pressure drop at the nozzle and for control of the flow rate for the entire system is considered adequate and applicable for this sampling practice.

3.2.2 Other Equipment

- Stop Watch
- Disposable Gloves
- Trash bag for disposing of wipes, gloves
- 50 cm long x 3 cm diameter brush for decontamination of sampling train
- 500-mL squeeze bottle with 0.01 M HCl
- Wipes
- Masking tape and marking pen
- Sieve 150 µm mesh; #100 sieve must be either stainless steel or plastic
- Analytical balance accurate to 0.1 g; weighing range of 0.1 mg to 1000g
- Template (4 ft^2)

3.2.3 Reagents and Materials

All chemicals used for decontamination must be reagent grade or better.

3.3 Preparation and Calibration

Preparation - Clean the wheels and nozzle lip with a clean laboratory tissue immediately before sampling. The sampling train shall be inspected to ensure that it has been cleaned and assembled properly. The sampling train shall be leak-checked prior to sampling. This can be accomplished by placing a mailing envelope or a piece of cardboard beneath the nozzle and switching on the suction source. The flow Magnehelic gage should read 5 Pa $(0.02 \text{ in. } H_2O)$ or less. If any leakage is detected, the system shall be inspected for the cause and corrected before use.

Calibration-The sampling strategy described in this practice does not have any calibrated flow devices other than the cyclone and the Magnehelic gages. The cyclone used for the separation of the particles must be designed to give proper separation at varying flow rates throughout the sampling range of the system. The pressure gages and any other devices (that is, temperature gage) used for testing purposes should be calibrated against a primary standard. Adjust the flow rate and nozzle pressure drop to values that approximate those given in Table 1.

Pressure Gages – Pressure gages shall be calibrated against an inclined manometer or other primary standard at the beginning of each day. One means of checking a Magnehelic gage is to set a flow rate through the sampling system with a manometer and then switch to the Magnehelic gage. If the difference in the readings is more than 3%, the gage is leaking or is in need of repair or calibration. This should be done at two different flow rates when checking the gage.

3.4 Sample Label and Field Data Sheet

Before beginning the dust collection protocol within a residence, first attach a pre-made site-specific sample identification label to a clean dust collection bottle and attach the bottle to the sampling device. Then attach the corresponding pre-made sample identification number to the field data sheet for that sample (see SOP ISSI-VBI70-01 Revision 0). This field data sheet is presented in Figure 2. On the data sheet, fill in the appropriate information on the sampling team, date, residence address, etc. As sampling progresses, record the location of each template collected on the field data sheet.

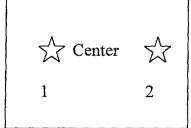
3.5 Sampling Locations within the Residence

A single composite of dust will be collected at each residence. This composite will be composed of dust collected from a number of sub-sampling locations, identified as below. All sub-samples will be collected in rooms or other living areas ("living spaces") where the residents are most likely to be exposed. This includes bedrooms, family and/or television rooms, kitchens, hallways and entryways.

In most cases, two templates will be collected per living space. Thus, the total number of sub-samples collected within a residence will be dependent upon the number of living spaces available. In the case where a residence has more than 10 living spaces, only 1 template per living space will be collected. This approach is recommended so that 20-30 sub-samples are not collected for a large residence.

Sub-sample locations within a living space (living space sample points) should focus on areas with the greatest potential for exposure. This is typically along the center axis of the living space. Corners of rooms, areas beneath furniture, etc., are not likely to be high exposure areas (even if especially dusty) and will not be sampled. A typical pattern of template locations within a living space is illustrated below:

Living Space



If obstructions are present at locations described above, the sub-sample location may be off-set accordingly, the new location noted in the field logbook and sample collected in accordance with this SOP.

3.6 Sampling Procedure

At each sub-location within the house to be sampled (see below), place the template on the sampling surface. If needed, use masking tape to temporarily hold the template to the surface so the template does not move during sampling. Turn on the vacuum and place the nozzle in one corner of the sampling area, then adjust the flow rate and pressure drop according to the type of surface. For hard surfaces or level loop carpet, the flow rate should be adjusted to at least 7.8 L/s (20 cfm), and the nozzle drop should be at least 2.2 kPa (9 in. H₂O). For plush or shag carpet, the flow rate must be at least 9.5 L/s, and the nozzle pressure drop must be at least 2.5 kPa (10 in. H₂O). The two factors that affect the efficiency of the sampling system are the flow rate and pressure drop at the nozzle. The pressure drop at the nozzle is a function of the flow rate and distance between the surface and the nozzle flange.

Begin sampling by moving the nozzle along one edge of the sampling area. Move the nozzle at approximately 0.5 m/s back and forth <u>four times</u> along the edge. Then move the nozzle inward a distance equal to the effect sampling width of the nozzle and make four passes parallel to the edge of the template. Repeat this strip-by-strip collection pattern until the entire template area has been covered.

Switch off the vacuum and move to the next sampling sub-location within the residence. Repeat the process at each sub-location. When all sub-locations within the residence have been sampled, the catch bottle can be removed, labeled, and capped for storage and analysis.

3.7 Decontamination - Sampler Cleaning

After all sub-samples have been collected at a residence, the sampling equipment must be thoroughly decontaminated before beginning sampling at the next residence. With the sample bottle removed and safely stored, open the flow control valve to maximum flow, tip the sampler back so that the nozzle is approximately 5 cm (2 in.) off the floor, and switch the vacuum on. Place a hand covered by a rubber glove on the bottom of the cyclone and alternate closing and opening the cyclone for 10 seconds to free any loose material adhering to the walls of the cyclone and tubing. It is not necessary to catch this small amount of dust, as it is usually much less than 1% of the collected sample.

Remove the sampler to a well-ventilated cleaning area free of dust. Remove the cyclone and elbow at the top of the nozzle tubing from the sampler. Use a 50-cm long by 3-cm diameter (20 by 1.25-in.) brush to clean the nozzle, and clean all related items up to and

including the cyclone and catch bottle with the hydrochloric acid solution that has been certified lead and arsenic free. The total amount of dust removed in the air and wet cleaning is usually much less than 1% of the collected dust. The air and wet cleaning is performed to prevent contamination from passing from one sample to another. After every 20 properties, an equipment blank will be collected. Equipment blank sample collection is described in the QC section (Section 6.2).

3.8 Prevention of Cross-Contamination

The following work practices should be followed to prevent cross-contamination of samples:

- Avoid disturbing and tracking dust from one location to another by identifying and
 clearly marking all sampling locations upon arrival at the sampling site, avoiding
 walking through or over any of the marked sampling location areas, and instructing
 field teams members to pull on new disposable shoe covers upon each entry into the
 building (this is especially significant if field teams have been walking through
 known exterior contamination sources).
- Use a new pair of powderless gloves at each sampling location.
- Inspect all sampling equipment for cleanliness prior to collection of each sample.
- Do not open sample collection containers until needed to collect each sample.
- Immediately remove and dispose of gloves when sampling is complete.

5.0 SAMPLE STORAGE AND ANALYSIS

5.1 Sample Storage

After collection of the sample in the catch bottle, the bottle should be tightly capped, the security of the sample identification number checked, and the bottle placed in an appropriate storage container. Storage at ambient temperature for up to 180 days is appropriate for samples that will be analyzed only for metals.

5.2 Sample Preparation

Before analysis, each dust sample will be sieved to removed large non-dust material (hair, fibers, objects, etc.). Sieve the samples thorough a #100 mesh screen to isolate particles that are 150 μ m or smaller. After sieving, weigh the sieved material to an accuracy of \pm 0.1g. This weight will be reported by the laboratory so that loading data may be determined.

5.3 Sample Analysis

The analytes of interest for indoor dust are arsenic and lead. Because the mass of dust collected from a residence is often too low to support reliable quantification by XRF techniques, samples will be digested using nitric acid (SW-846 method 3050 or 3051) and analyzed using standard USEPA protocols via either graphite furnace atomic absorption (GFAA) or Inductively Coupled Plasma/Mass Spectrometry (ICP/MS), providing the following detection limits are achieved:

Arsenic 1.0 mg/kg Lead 5.0 mg/kg

6.0 FIELD QUALITY ASSURANCE/QUALITY CONTROL

Adherence to quality assurance/quality control (QA/QC) procedures is an important part of field sample collection. Field QA/QC procedures include documentation requirements, and field QC samples.

6.1 Documentation Requirements

All field documentation requirements are included in the Indoor Dust Data Sheet (see Figure 2). Each sampling team must ensure that all required items are recorded on this field data sheet, that the sample number is firmly affixed, and that any deviations from the SOP are noted on the sheet.

6.2 Field QC Samples

<u>Field blanks</u>. Field blank samples are used to identify any potential systematic contamination present in the catch bottle or wipes and handling of samples during field collection and laboratory analysis activities. Field blanks should be collected in the same manner used to collect field samples with the exception that the vacuum sampling nozzle is pointed away from the floor and air is drawn through the catch bottle. Remove bottle, cap, and record on a field data sheet.

<u>Blind Standard (Reference Material) Samples.</u> Blind standard will be submitted to the laboratory to determine the accuracy of metals analysis using this sample collection method. The these QC samples will be submitted blindly to the laboratory at a frequency of 20% (1 blind standard per 20 field samples).

7.0 REFERENCES

American Society for Testing and Materials (ASTM). 1994. Standard Practice for Collection of Dust from Carpeted Floors for Chemical Analysis. ASTM Designation: D5438-93. September.

Bornschein. 1989. Midvale Community Lead Study, Appendix B: Quality Assurance Plan.

USEPA. 1995a. Residential Sampling for Lead: Protocols for Dust and Soil Sampling. Final Report. U.S. Environmental Protection Agency, Office of Pollution Prevention and Toxics. EPA 747-R-95-001.

USEPA. 1995b. Sampling House Dust for Lead. Basic Concepts and Literature Review. U.S. Environmental Protection Agency, Office of Pollution Prevention and Toxic Substances. EPA 747-R-95-007.

USEPA. 1996. Sampling Manual for IEUBK Model. Prepared by Roy F. Weston. Document control number 4800-045-0019.

Roberts, JW, et. al. 1991. A Small High Volume Surface Sampler HVS3 for Pesticides, and Other Toxic Substances in House Dust, Paper Number 91-150.2, 84th Annual Meeting, Air & Waste Management Association, Vancouver, British Columbia, June 16-21, 1991.

Roberts, JW and MG Ruby. 1989. Development of a High Volume Surface Sampler for Pesticides, U.S. Environmental Protection Agency Report No. EPA 600/4-88/036, Research Triangle Park, N.C. January 1989.

Stamper, VR, et. al. 1990. Development of a High Volume Small Surface Sampler for Pesticides and Toxics in House Dust, Research Triangle Park, N.C. June 1990. Included in supporting data, which are on file at ASTM Headquarters Request RR:D22-1010.

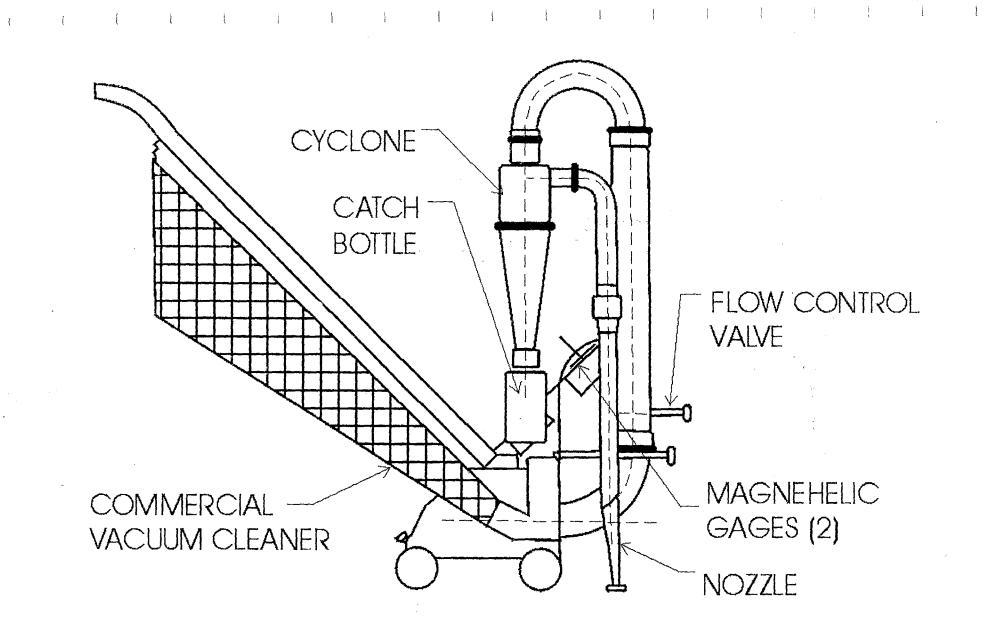


Figure 1: Dust sampler using a commercial vacuum cleaner as the suction source.

FIGURE 2 INDOOR DUST DATA SHEET



PHASE:	3	
MEDIUM:	INDOOR DUST	
SOP:	ISSI-VB170-03 Revision 0	And the second second
DATE:	<u> </u>	-
SAMPLE TEAM ID:		
LOCATION:		·
	House#	Street Name
CLASS:	FS	(Field Sample)
	EB	(Equipment Blank)
	FB	(Field Blank)
SAMPLE TYPE:	COMP	
TEMPLATE SIZE:	4 ft ²	

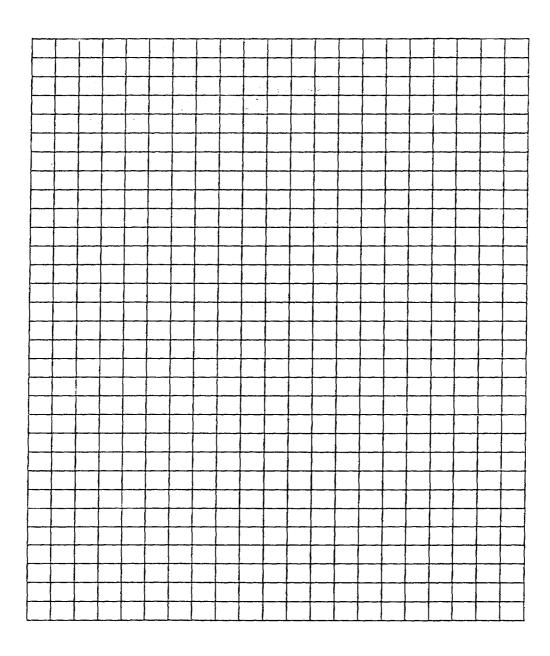
TEMPLATE COLLECTION LOCATIONS:

Number	Living Area (a)	Surface Type (b)	Notes
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			
18			

(a) Living Area Codes:
BR = bedroom
FR = family room / living room
K = kitchen
D = dining / eating area
H ≕hall way
E = entry way
O = other (note which)

(b) Surface Types:
H = hard (linoleum, stone, wood, etc.)
S = soft (carpet, rug, etc.)
O = other (note which)

Field Diagram:



Samples Collected by:		
	Signature	Date
Logbook Page Reviewed by:		
	Signature	Date

Figure 3

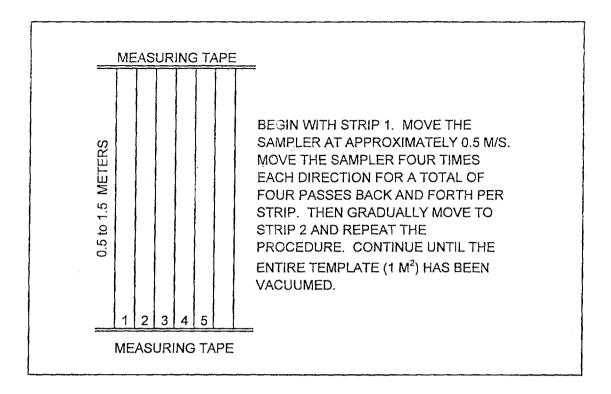


TABLE 1 Approximate Values for Flow Rate and Nozzle Pressure Drop

Carpet Type	Flow Rate	Nozzle Pressure Drop
Plush	9.5 L/s (20 CFM)	2.2 kPa (9 in. H₂0)
Level loop	7.8 L/s (18 CFM)	2.5 kPa (10 in. H ₂ 0)

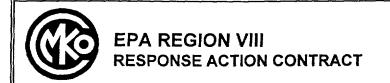
TABLE 2 Sampling Efficiency Using Modified Laboratory Test Method F 608^a

Parameters -	Carpet Type	
raiailleters -	Plush	Level Loop
Flow rate (L/s)	9.4	7.6
Delta P(kPa) ^b	2.3	2.5
Mean % of mass collected in cyclone	69.5	66.8
Standard deviation	1.2	2.8
Number of tests	1	3

^a Carpet dust loading was 15.9 g/m².

^b Pressure drop at nozzle.

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Author:		Date
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TECHNICAL STANDARD OPERATION PROCEDURE PROPERTY ACCESS

MANAGEMENT APPROVAL

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	PROGRAM MA	NAGER	-	DATE	_
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PAGE:2 OF 5

SOP: PROPERTY ACCESS

1.0 PURPOSE

The purpose of this SOP is to provide procedures and guidelines to the Morrison Knudsen personnel assigned to the VB/I-70 project and their subcontractors regarding requesting consent for property access, conditions that limit access, and proper conduct when working on private property.

2.0 SCOPE

This procedure covers activities associated with outdoor and indoor sampling activities on private properties targeted for sampling under this project.

3.0 RESPONSIBILITIES

All **Field Personnel** are responsible for implementing this procedure as specified, recording communications with property owners, ensuring personal safety, and notifying the Field Supervisor of any problems or concerns.

The **Field Supervisor** is responsible for training field personnel to this procedure, emphasizing the need for safe and professional conduct of all field personnel, reviewing communication records, and notifying the Site Manager of any problems or concerns. The Field Supervisor will also provide information to field personnel identifying properties for which access has been received and those for which access is to be requested.

The **Site Manager** is responsible for ensuring that proper resources are provided for training to this procedure and maintenance of safe working conditions, and requesting support from EPA in responding to access problems or concerns.

4.0 REQUIREMENTS

4.1 General

Only those properties for which written consent for property access has been provided by the owner will be sampled. To the extent possible, written consent for access will be obtained in advance of the sampling program. Field personnel will attempt to gain written consent for access for additional properties in the course of the sample program. Field personnel will work in crews of at least two at all times.

4.2 Conditions Limiting Access



REVISION: 0

PAGE:3 OF 5

SOP: PROPERTY ACCESS

Providing a safe working environment is MK's highest priority. The field personnel must be cognizant of all conditions existing at an individual property, and are required to immediately leave the property if there are any safety concerns, including any of the following conditions:

- Dog or other potentially dangerous animal is unrestrained or inadequately restrained
- Resident or owner requests that personnel leave
- Resident, owner or other individual verbally threatens or harasses any personnel
- Resident or owner requires samples be collected in a location or manner contrary to the approved procedure
- Signs of any criminal activity are observed
- "Keep Out" or equivalent sign is posted and written access has not been received
- Gates of any height are locked
- Potential physical, biological or chemical hazards exist

4.3 Request for Property Access

- 4.3.1 Field personnel will request access from selected properties as directed by the Field Supervisor.
- 4.3.2 Personnel will possess proper identification and the following information in English and Spanish:
 - EPA Phase III sampling fact sheet(s)
 - Cover letter sent to property owners
 - Access agreement
 - Letter from community representatives
- 4.3.2 Personnel will knock or ring the door bell at the front door of the home, provided that none of the Conditions Limiting Access listed above exist.
- 4.3.3 If the resident does not answer, an EPA-approved bilingual notice will be left at the door.
- 4.3.4 If the resident answers, personnel should ask to speak to a parent if a minor answers the door, and should communicate the following:
 - Name and affiliation
 - Purpose of visit
 - Inquire as to whether the adult resident is the owner
 - If no, ask whether they would like written information to forward to the owner
 - If yes, explain that sampling is ongoing in their area, provide the written information, provide an opportunity to sign and submit the access agreement at that time, and ask owner how long they have owned the property



REVISION: 0

PAGE:4 OF 5

SOP: PROPERTY ACCESS

 Answer any commonly asked questions and if needed explain that other questions will be forwarded to EPA and that someone will contact them with a response

- 4.3.5 If the resident signs the access agreement, the field personnel will verify the name against the ownership records (1998 tax assessor records). Proceed to collect samples if any of the following apply:
 - The owner name matches either the owner or co-owner listed in the records
 - The owner stated that property was purchase within the last year
 - The name is not listed in the ownership records
- 4.3.6 If the resident signs the access agreement but 4.3.5 does not apply, additional measures may be taken to confirm receipt of the property owners' consent.
- 4.3.7 Personnel will record the following information for each property in an access logbook:
 - Date and time
 - Property address
 - Whether adult resident was home
 - Whether agreement was signed
 - Owner name(s) listed in ownership records
 - Summary of communications
 - Inquiries, complaints and items requiring EPA response

4.4 Personnel Conduct

- 4.4.1 Field personnel will take all available measures to prevent damage to private property, to restore the property to its previous condition, and to be respectful of residents. All trash, equipment and materials used by field personnel will be removed from the property upon completion of sampling.
- 4.4.2 The following activities are not permitted on residential properties:
 - Unauthorized parking
 - Loitering or spending break time
 - Smoking
 - Offensive language or behavior
 - Trespassing over locked gates
 - Unauthorized entry to home or exterior structures
 - Disturbing any area or vegetation except as required by the sampling procedures

5.0 ATTACHMENT

Property Access Cover Letter, Agreement, and Letter from Community Representatives



REVISION: 0

PAGE:5 OF 5

SOP: PROPERTY ACCESS

ATTACHMENT

PROPERTY ACCESS COVER LETTER, AGREEMENT AND LETTER FROM COMMUNITY REPRESENTATIVES



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY REGION 8

999 18th STREET - SUITE 500 DENVER, CO 80202-2466

Ref: 8EPR-SR

June 11, 1999

Dear Property Owner/Resident:

The Environmental Protection Agency (EPA) is investigating the soils in the Swansea, Elyria, Cole, Clayton, and Globevillle neighborhoods of Denver. You may be aware that in the spring and summer of 1998, we conducted a study in these neighborhoods by collecting soil samples from over 1300 residential yards. Soil was tested for arsenic and lead content. The majority of yards sampled have low levels, but some had levels high enough to warrant replacement of the soil in the yard.

This summer, EPA will be collecting and testing soil from residential yards that were not tested last year. Our study boundaries are 56th Avenue to the north, Martin Luther King Boulevard on the south, the South Platte River on the west, and Colorado Boulevard on the east. We will also include a small area in Globeville that is west of I-25 (to Fox Street) and south of I-70. We'd like to sample every residential yard in the study area. This information will be important for EPA's use in looking at patterns of lead and arsenic levels and for predicting potential health risks associated with contact with soil. We also think this is important information for every homeowner to have to ensure protection of your health and that of your family.

EPA requests your written permission to conduct this work on your property. Your name and signature will not be publicly available. Soil collection and testing is free. If you are willing to volunteer to have your soil sampled and tested, please follow these steps:

- (1) PLEASE READ AND SIGN THE ATTACHED "ACCESS AGREEMENT."
- (2) PLACE THE SIGNED ACCESS AGREEMENT IN THE ENCLOSED POSTAGE PAID ENVELOPE ADDRESSED TO EPA'S CONTRACTOR, MORRISON KNUDSEN, AND DROP IT IN THE MAIL.

You don't need to be home when the samples are collected but if you would like to be, we will attempt to schedule sampling when you are there. If you are renting the property, please ask the owner to sign the agreement. Soil samples will be sent to a laboratory for testing. This work may take up to three months. When they are available, EPA will mail individual results to each property owner.

If you have questions about this study, please don't hesitate to call Pat Courtney at (303) 312-6631, or Ted Fellman at (303) 312-6119. Thank you very much for your cooperation.

Sincerely,

Bonnie Lavelle Remedial Project Manager



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY REGION 8

999 18th STREET - SUITE 500 DENVER, COLORADO 80202-2466

ACCESS AGREEMENT

	PROPERTY:		
	onmental Protection Agency (EPA) staff and EPA's a erty identified above for the purpose of collecting soil at no cost to me.	~	
the north Denver	his soil testing is part of an investigation of possible area. EPA is conducting this investigation as part nvironmental Response, Compensation and Liability	of its responsibilities under	the
Print Name		Date	
Signature		Phone Number	
I would like	ollowing if applicable: EPA to provide me with a portion of the sample, called	d a "split sample," that I may l	iave
	my own expense.		
	my own expense. Destions, please contact Ted Fellman at (303) 312-61 Corporation (EPA's contractor) at (303) 948-4693.	119, or Marta Valentine from	the

PLEASE SIGN AND RETURN THIS ACCESS AGREEMENT TO OUR CONTRACTOR IN THE ENCLOSED PREPAID ENVELOPE. Soil sampling will take about I hour. The owner or resident need not be present. If you would like to be notified when we plan to sample your property, please state so in the Comments section and provide your phone number. Also, pet owners are asked to provide a phone number so that if necessary we may schedule the sampling at a time when the pet will be indoors or restrained. Thank you for participating in this important study of your neighborhood.

NOTE: If you are <u>not</u> the current property owner, and you are not a renter who wishes to forward this request to the owner, please state so in the Comments section and return this agreement unsigned.



LA AGENCIA DE PROTECCIÓN DEL MEDIO AMBIENTE DE LOS ESTADOS UNIDOS REGIÓN 8 999 18TH STREET – SUITE 500 DENVER, COLORADO 80202-2466

Ref: 8EPR-SR

junio 11, 1999

Propietario/Residente:

La Agencia de Protección del Medio Ambiente (EPA), actualmente esta haciendo estudios de suelo en los vecindarios de Swansea, Elyria, Cole, Clayton y Globeville de la ciudad de Denver. Ud puede haber notado que durante la primavera y el verano del año 1998, se hicieron estudios en estos vecindarios coleccionando muestras de suelo de mas de 1,300 patios de casas residenciales. Los suelos fueron examinados para poder determinar el contenido de arsénico y plomo presente. En general, la mayoria tenían un contenido bajo o minimo, pero en algunos casos el contenido estaba suficientemente alto, el cual requería que el suelo del patio existente sea reemplazado.

Este verano, EPA estara coleccionando muestras de patios residenciales que no fueron examinados el año anterior. Los limites para los estudios estan entre la Avenida 56 al norte, Martin Luther King Boulevard al sur, el rio South Platte al oeste y Colorado Boulevard al este. Al igual estara incluida una pequeña porcion en Globeville la cual esta al oeste de la autopista I-25 (a la calle Fox) y al sur de la autopista I-70. El plan es de asegurar que cada una de las residencias incluidas en el estudio sean examinadas. La información obtenida sera muy importante para que EPA pueda establecer niveles de arsénico y plomo existentes y al mismo tiempo poder predecir el potencial de riesgos a la salud debido al contacto con el suelo existente. Al igual, creemos que la información sea muy importante para toda familia residente, asegurando que cada una tenga la debida protección personal y de su familia.

EPA pide el permiso en escrito para realizar los examenes de suelo en su propiedad. Su nombre o firma no seran hechos publicos. La colección de suelos y los examenes de los mismos es gratis. Si usted esta dispuesto acceder voluntariamente al estudio de suelos, por favor siga los siguientes pasos:

- (1) Por favor lea y firme el formulario incluido "Acuerdo de Acceso."
- (2) Coloque el formulario firmado en el sobre adjunto con la dirección de la firma de contratos para EPA, Morrison Knudsen y deposítelo en un buzon de correo (no se necesita una estampilla).

Ud no necesita estar presente en su domicilio durante la colección de muestras de suelo, pero si desea estar presente, trataremos de marcar un horario que asegure su presencia. Si usted esta alquilando la propiedad, por favor pida que el dueño de la casa firme dicho formulario. Las muestras de suelo coleccionados, seran enviados a un laboratorio para hacer el debido análisis. El análisis tomara unos tres meses y al cabo de estos, los resultados seran enviados a cada dueño de casa.

Si tiene algunas preguntas correspondientes a los estudios, por favor llame a Ted Fellman al número (303) 312-6119. Se le agradece mucho por su coperación.

Aténtamente,

Bonnie Lavelle Gerente del Proyecta

Adjuntos



LA AGENCIA DE PROTECCIÓN DEL MEDIO AMBIENTE DE LOS ESTADOS UNIDOS REGIÓN 8

999 18TH STREET - SUITE 500 DENVER, COLORADO 80202-2466

ACUERDO DE ACCESO

Para la propiedad indicada al otro lado de la hoja

Permitiré al personal de la EPA y a sus representantes autorizados tener acceso a mi propiedad para el propósito de recolectar muestras di tierra. Entiendo que este servicio se proporcionará sin ningún costo para mí.

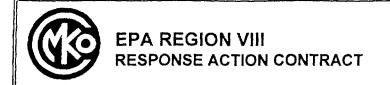
Entiendo también que este análisis de suelos forma parte de una investigación sobre la posible contaminación por metales de los suelos del norte de Denver. La EPA realiza esta investigación como parte de sus responsabilidades, de acuerdo con las disposiciones de la "Comprehensive Environmental Response, Compensation and LíabilityAct" (Respuesta Ecológica Integral, Ley de Responsabilidades y Compensaciones), una ley también conocido como "Superfondo."

Nombre Impreso		Fecha	
Firma		Telefono	
Por fav	or marque el partafo si le corresponde:		
Pido que la EPA me deje una porción de las de muestras coleccionadas en mi p llámadas "split sample." Entiendo que el costo del analisis sera mio.			
Si tiene	algunas preguntas, por favor llame a Ted Fellm	an (303) 312-6119.	
Sus Co	mentarios:		

POR FAVOR FIRME ESTE FORMULARIO DE ACUERDO DE ACCESO Y ENVIELO A NUESTRO CONTRATISTA EN EL SOBRE INCLUIDO (no se necesita una estampilla). El trabajo tomara aproximadamente una hora. Ud. no necesita estar presente en su domicilio durante la colección de muestras de suelo. Si desea ser notificado, indiquelo bajo la sección de comentarios y incluya su teléfono. Igualmente, personas dueñas de animales domésticos deberan dejar un número de teléfono en caso que sea necesario fijar la hora de colección a un tiempo mas propio asegurando que el animal este encerrado o amarrado. Otra vez, muchas gracias por participar en este estudio para el bien de la vecindario.

Nota: Si usted no es deuño de la presente propiedad, y no desea mencionar este trabajo a los dueños, por fabor indiquelo en la parte marcada para comentarios y devuelva estos formularios sin firmar.

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TECHNICAL STANDARD OPERATION PROCEDURE CHAIN OF CUSTODY AND SAMPLE HANDLING

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PAGE:2 OF 5

SOP: CHAIN OF CUSTODY AND SAMPLE HANDLING

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide instructions to MK and assigned to the Vasquez Boulevard/I-70 project, and their subcontractors, on maintaining and documenting chain of custody (COC) and on containing, preserving, and packaging samples for shipment to off-site laboratories.

2.0 SCOPE

This procedure covers activities associated with maintaining and documenting chain of custody and environmental sample handling. This procedure does not cover activities associated with submitting samples for analysis under EPA's Contract Laboratory Program.

3.0 REFERENCES

MK Engineering Standard 3.4, Sampling Handling Requirements

EPA Method 6010: Inductively Coupled Plasma Atomic Emission Spectroscopy, SW-846

4.0 RESPONSIBILITIES

- 4.1 The **Sampler** is responsible for collecting samples in appropriate containers, labeling sample containers and maintaining and documenting sample custody until the samples are relinquished.
- 4.2 The **Field Supervisor** is responsible for review of sample labeling, chain of custody documentation, and packaging of samples for shipment.
- 4.3 The **Project Chemist** will be responsible for verifying implementation of this procedure through surveillance and maintaining records.



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PAGE:3 OF 5

SOP: CHAIN OF CUSTODY AND SAMPLE HANDLING

4.4 The Site Manager will be responsible for ensuring that records are properly maintained and that personnel are trained to this procedure.

5.0 REQUIREMENTS

5.1 Chain of Custody Procedures

Chain of custody must be maintained at all times and documented on a COC form. A sample is in an individual's custody if:

- It is in his/her possession
- It is in his/her view, after being in their possession
- It was in his/her possession and he/she either locked it or placed it in a sealed container to prevent tampering
- It is in a designated secure area
- 5.1.1 Chain of custody forms shall be used for all samples submitted to either the on-site laboratory or an off-site laboratory. An example COC for an MK contract lab is presented as Attachment 1.
- 5.1.2 Each sample shall be entered on the COC at the time of sample collection by the Sampler(s). The following information shall be entered:
 - Project identification
 - Sample identification number
 - Date and time sampled
 - Sample media
 - Required analyses
 - Number of containers collected for the sample
- 5.1.3 Each person on the sampling team shall sign the COC in the Samplers Signature box. When samples are relinquished, one of the sampling team members will sign the "Relinquished By" signature block at the bottom of the COC and enter the date and time. The person receiving the samples will sign the "Received By" signature block.



REVISION: 0

PAGE:4 OF 5

SOP: CHAIN OF CUSTODY AND SAMPLE HANDLING

5.1.4 One copy of the COC will be retained along with corresponding airbills and provided to the Project Chemist for review and filing.

5.1.5 Custody seals will be placed on all containers used to ship samples to an off-site laboratory, and also will be used whenever the samples are not in view or in a secured area. The seals must be placed so that is would not be possible to tamper with the sample without disturbing the seal.

5.2 Sample Containment, Preservation and Holding Times

- 5.2.1 Samples will be placed in containers compatible with the analytical request and laboratory requirements. Soil samples collected for metals analysis by XRF will be collected in plastic, sealable bags. Soil samples collected for metals analysis at an off-site laboratory may be collected in plastic, sealable bags or in glass jars. Dust sample filter cartridges for metals analysis will be placed in plastic, sealable bags. Sealed plastic bags will be checked to verify complete closure of the seal.
- 5.2.2 Soil and dust samples will not require any preservation. Samples should be handled and stored to maintain integrity and prevent damage to the container.
- 5.2.3 Soil and dust samples submitted for analysis by ICP Method 6010 will have a maximum holding time for analysis of 6 months following sample collection.

5.3 Sample Packaging and Shipping Procedures

- 5.3.1 Samples will be packaged so as to minimize the possibility of container breakage, and to provide containment in the event of container breakage or leaking. Any samples in glass containers for off-site shipment will be packaged using bubble wrap or equivalent packing materials.
- 5.3.2 All samples shipped to an off-site laboratory will be contained in a plastic cooler with packing material, if necessary, to prevent excessive agitation of the contents.



REVISION: 0

PAGE:5 OF 5

SOP: CHAIN OF CUSTODY AND SAMPLE HANDLING

5.3.3 A notation will be made in the "Received By" block on the COC form that a cooler was sealed for shipment via the carrier. One copy of the COC form will be retain, and the completed form will be verified against the cooler contents, placed in a sealable bag, and taped to the inside top of the cooler.

- 5.3.4 All coolers will be securely taped closed, sealed with a minimum of two signed custody seals and labeled with a completed air bill prior to shipment.
- 5.3.5 Samples that are identified as possible dangerous goods will be shipped in accordance with appropriate DOT regulations for hazardous materials.

6.0 ATTACHMENTS

Attachment 1 - Example Chain of Custody Form

ATTACHMENT 1

(Example Chain of Custody Form)

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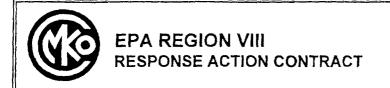
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TECHNICAL STANDARD OPERATION PROCEDURE FIELD DOCUMENTATION

MANAGEMENT APPROVAL

SITE MANAGER	DATE
PROGRAM MANAGER	DATE

REV.	DATE	REVISION DESCRIPTION
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ORIGINATOR: Marta Valentine



NUMBER:

REVISION: 0

PAGE:2 OF 3

SOP: FIELD DOCUMENTATION

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide instructions for maintaining field documentation for Vasquez Boulevard/I-70 field investigations.

2.0 SCOPE

This procedure covers logs and data sheets maintained by the sampling personnel.

3.0 RESPONSIBILITIES

The **Sample Lead** will be responsible for maintaining the sample documentation during sampling activities and providing the documentation to the Field Supervisor.

The **Field Supervisor** will be responsible for receiving, reviewing, maintaining the sample documentation records. He/She also will train the sampling personnel to the requirements of this procedure. The Field Supervisor will retain copies of the records and forward original documents to the Site Manager.

The **Site Manager** will ensure that records are maintained and filed properly and that all personnel are trained to this procedure.

4.0 REQUIREMENTS

Field documentation consists of field data sheets, field logbooks, and calibration logs. A hard bound logbook will be used by each sample crew. Information recorded in the hard bound logbook may be supplemented by loose leaf data sheets, in accordance with the following procedures:

Pre-number pages and annotate any unused pages. Make entries as events occur
throughout the day's activities. Use black permanent ink pens. Do not use white-out or
erase; rather, line out, initial and date any errors.



REVISION: 0

PAGE:3 OF 3

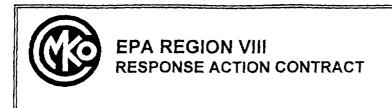
SOP: FIELD DOCUMENTATION

- Log entries using a 24-hour system.
- Report operational information:
 - Progress
 - Location
 - Site Conditions
 - Site Sketch
 - Workers in attendance
 - Any unplanned events or deviation from the project procedures
 - Communications with non-MK or subcontractor personnel
- Field documents constitute legal documents. All entries should be factual and objective, and without personal feelings or opinions. Entries related to any concerns, errors made, or omissions are legitimate entries.
- Each page must be numbered, initialed and dated.
- Pages or portions of pages not used should be lined out and initialed/dated.
- In the event that field instrumentation is required, this equipment will be calibrated according to manufacture's instructions. Calibration records and instrument identification will be documented in the field logbooks or calibration logs for each sample team.

TECHNICAL STANDARD OPERATING PROCEDURE --DRAFT--

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REVIEWS: TEAM MEMBER EPA Region 8	SIGNATURE/TITLE	<u>DATE</u>

Technical Standard Operating Procedure Morrison Knudsen SOP NO: ____ Revision No:_0__ Date: 6/99



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PAGE: 1

TECHNICAL STANDARD OPERATION PROCEDURE DECONTAMINATION

MANAGEMENT APPROVAL

SITE MANAGER	DATE
PROGRAM MANAGER	DATE

REV.	DATE	REVISION DESCRIPTION

ORIGINATOR: Marta Valentine



REVISION: 0

PAGE:2 OF 3

SOP: DECONTAMINATION

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide instructions for decontamination of sampling equipment and field personnel. Decontamination is necessary to protect personnel and to minimize the potential for cross-contamination of samples. This procedure is to be used by MK employees assigned to the Vasquez Boulevard/I-70 project and their subcontractors.

2.0 SCOPE

This procedure covers activities associated with decontamination of sample equipment and personnel. Additional requirements for personnel decontamination may be specified in the Site Health and Safety Plan.

3.0 RESPONSIBILITIES

All Field Personnel will be responsible for performing personal and equipment decontamination after sampling at each location and at the end of the day in accordance with these procedures.

The **Field Supervisor** will be responsible for training field personnel in appropriate decontamination procedures as well as verifying implementation of this procedure through surveillance.

The **Site Manager** will be responsible for ensuring that all personnel are trained to this procedure.

4.0 DECONTAMINATION

4.1 Personnel will remove disposable gloves following collection of each sample. Gloves will contained in a plastic bag and disposed as municipal waste. All personnel and clothing will be inspected following sample collection at each property and, if necessary, decontaminated to remove any potential harmful substances that may have adhered to them. Disposable, pre-moistened wipes will be available for personnel to wash their face and hands.



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SOP: DECONTAMINATION

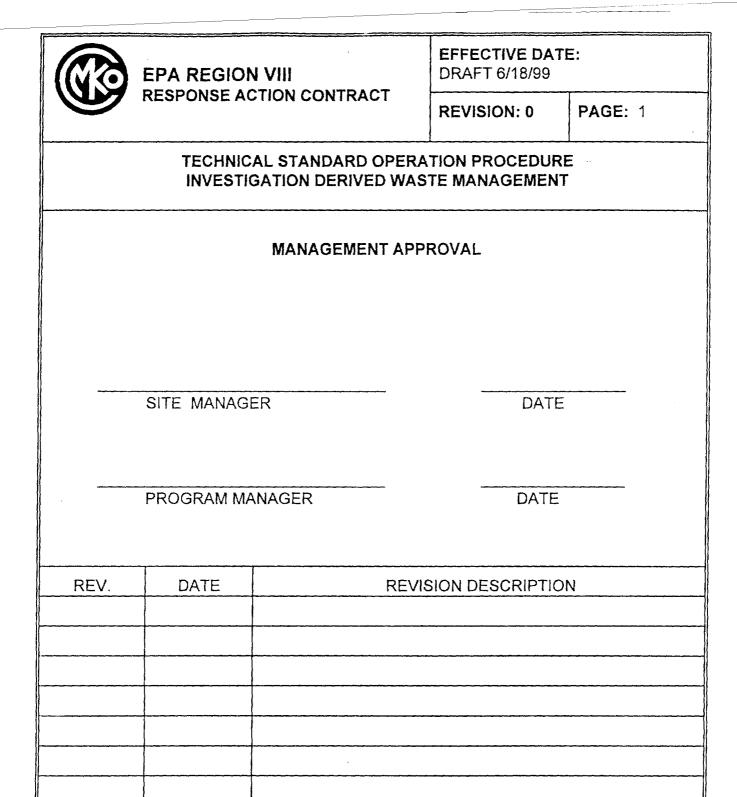
- 4.2 The equipment used for sampling, including hand augers, sieves, bowls and trowels, will be decontaminated between samples collected for separate composites, between samples collected for discrete sampling and analysis, and following the last sample collection daily. Sampling equipment will be decontaminated by the following procedure:
 - Equipment will be decontaminated immediately following sample collection at the location/property from which the sample was collected
 - Equipment will be washed with a low- or non-phosphate detergent and tap water using a brush as necessary
 - Equipment will be triple rinsed with deionized water
 - After decontamination, equipment and tools will be protected by placing them in clean containers and taking care not to allow contact with surface soils
- 4.3 Decontamination rinsate will be disposed in accordance with the Technical Standard Operating Procedure for Investigation Derived Waste Management.

TECHNICAL STANDARD OPERATING PROCEDURE --DRAFT--

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Technical Standard Operating Procedure Morrison Knudsen

SOP NO: ____ Revision No:_0__ Date: 6/99



ORIGINATOR: Marta Valentine



REVISION: 0

PAGE:2 OF 5

SOP: INVESTIGATION DERIVED WASTE MANAGEMENT

1.0 PURPOSE

The purpose of this procedure is to describe the methods that will be used by Morrison Knudsen personnel assigned to the VB/I-70 project and their subcontractors to manage investigation derived wastes (IDW).

2.0 SCOPE

This procedure covers management of all IDW, including trash, soils, water, and personal protective equipment (PPE). Management procedures include waste collection, segregation, characterization, storage, shipping and disposal, as appropriate for each waste stream.

3.0 REFERENCES

Management of Investigation-Derived Wastes During Site Inspections, EPA/540/G-91/009

Code of Federal Regulations, Chapter 50, Parts 262 and 265.

4.0 DEFINITIONS

DOT: Department of Transporation IDW: Investigation Derived Waste PPE: Personal Protection Equipment

5.0 RESPONSIBILITIES

All Field Personnel will be responsible for managing IDW in accordance with this procedure.

The **Field Supervisor** will be responsible for training field personnel to the requirements of this plan, verifying its implementation, and generating and maintaining required records.

The **Site Manager** will be responsible for ensuring that personnel are properly trained and providing guidance for any special circumstances that may arise.

6.0 EQUIPMENT

- DOT compliant containers as specified in 40 CFR 265 Subpart I.
- Non-hazardous (and if necessary Hazardous) Waste Labels
- Spill Control Materials



REVISION: 0

PAGE:3 OF 5

SOP: INVESTIGATION DERIVED WASTE MANAGEMENT



REVISION: 0

PAGE:4 OF 5

SOP: INVESTIGATION DERIVED WASTE MANAGEMENT

7.0 REQUIREMENTS

7.1 General

All IDW will be managed in accordance with federal, state, and local rules and regulations. Personnel responsible for waste labeling, inspecting, profiling, manifesting, and transportation preparation will be trained per 29 CFR 1910.120 and 49 CFR 172.704.

7.2 Waste Types

Waste streams anticipated to be generated during the work activities include:

- Large fraction soils and vegetation
- Fine fraction prepared and analyzed soil
- Disposable gloves and other personal protection equipment (PPE)
- Decontamination rinsate generated at residential properties
- Decontamination rinsate generated at the field office/laboratory
- Trash
- 7.2.1 <u>Large Fraction Soils and Vegetation</u> The large fraction soils and vegetation will be separated from the fine fraction soils during sample preparation and sieving procedures. Large fraction soils or vegetation, including sod, generated at individual residential properties should be left at the property in the vicinity of the sample location(s). Large fraction soils or vegetation generated during sample preparation at the field office/laboratory will be contained and stored in drums pending profiling and disposal as described below.
- 7.2.2 <u>Fine Fraction Prepared and Analyzed Soil</u> Fine fraction soils will be generated at the field office/laboratory by the sample preparation process. Any portion of the prepared soils that are not archived will be contained and stored in drums pending profiling and disposal as described below.
- 7.2.3 <u>Disposable Gloves and PPE</u> Disposable PPE including gloves will be double bagged and disposed along with trash at a municipal landfill. Gloves that are grossly impacted by soils will be decontaminated prior to disposal.
- 7.2.4 <u>Decontamination Rinsate Generated at Residential Properties</u> Rinsate generated at individual properties from equipment or personnel decontamination will be disposed on the property at which the equipment was used, prior to leaving that property.
- 7.2.5 <u>Decontamination Rinsate Generated at Field Office/Laboratory</u> Rinsate generated at the field office/laboratory from equipment or personnel decontamination will be contained and stored in drums pending profiling and disposal as described below.



REVISION: 0

PAGE:5 OF 5

SOP: INVESTIGATION DERIVED WASTE MANAGEMENT

7.2.6 <u>Trash</u> - All trash generated during the project will be contained in plastic trash bags for pick-up and disposal by a municipal trash management company; unauthorized disposal of trash in trash recepticals that service City of Denver residents and businesses will not be permitted.

7.3 Contained Waste

- 7.3.1 <u>Containers</u> Soils and decontamination rinsate waste generated at the field office/laboratory will be contained in DOT-compliant drums in accordance with 40 CFR 265 Part I. Trash and PPE contained outdoors will be placed in a closed plastic trash receptical to prevent disturbance by animals and dispersion by wind.
- 7.3.2 <u>Labeling</u> All drummed IDW will be labeled as to its contents, source of material and the date on which waste accumulation begins. Non-hazardous waste labels will be used if appropriate. Additional labeling requirements specified in 40 CFR 262 and 265 Subpart I will be performed for waste that it determined to meet the criteria of a RCRA Hazardous Waste.
- 7.3.3 Storage All drummed IDW will be stored in a designated area and in a manner that minimizes the potential for container damage or personnel injury. Non-hazardous waste will be segregated from waste that is determined to meet the criteria of a RCRA Hazardous Waste. As a protective measure, hazardous waste will be stored in a secure (fenced), lined, bermed area, and will be subject to weekly inspections in accordance with 40 CFR 262. Water accumulating in the lined storage area after a precipitation event will be removed and contained with the non-hazardous rinsate waste.
- 7.3.4 <u>Profiling</u> All drummed IDW will be profiled using knowledge of the material and/or analytical data. Profile forms will be completed and submitted to the appropriate disposal facility as the basis of waste acceptance.
- 7.3.5 <u>Transporation and Disposal</u> Drummed IDW will be transported and disposed by transporters and facilities permitted to manage the profiled waste. All non-hazardous waste will be managed as industrial or special waste, and shipped under a non-hazardous waste bill of lading. Hazardous waste will be shipped to an EPA-approved RCRA Subtitle C facility under a RCRA Uniform Hazardous Waste Manifest, identified with the EPA Generator ID, and in accordance with all DOT requirements for shipping hazardous materials. A DOT Hazardous Material Registration must be provided by the transporter and accompany each hazardous material shipment. Disposal certification will be obtained from the RCRA Subtitle C facility.



REVISION: 0

PAGE:6 OF 5

SOP: INVESTIGATION DERIVED WASTE MANAGEMENT

7.4 IDW Log

A waste log will be developed and maintained to document the following information:

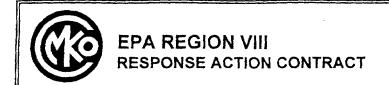
- Description of waste generated (e.g. soils, water)
- Classification of wastes (non-hazardous, hazardous, etc.) including EPA code as applicable
- Quantities of waste generated
- Type of waste storage container
- Dates of waste generation
- Manifest/Bill of Lading Numbers

8.0 RECORDS

- Waste Log
- Waste Profiles
- Non-Hazardous Waste Manifest/Bill of Lading
- RCRA Uniform Hazardous Waste Manifest, if needed
- Hazardous Waste Disposal Certification, if needed
- DOT Hazardous Material Registration, if needed

TECHNICAL STANDARD OPERATING PROCEDURE --DRAFT--

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TECHNICAL STANDARD OPERATION PROCEDURE XRF INSTRUMENT OPERATION

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PAGE:2 OF 11

SOP: XRF INSTRUMENT OPERATION

1.0 PURPOSE

The purpose of this procedure is to provide instructions to Morrison Knudsen (MK) personnel assigned to the VB/I-70 project and their subcontractors on the proper XRF operation protocol.

2.0 SCOPE

This procedure covers activities associated with intrusive sample analysis by utilizing an energy dispersive X-ray fluorescence spectrometry method (EDXRF). The procedure details operation of the KevexSpectrace QuanX instrument, which is a field portable, but not hand held, XRF.

3.0 REFERENCES

Spectrace, QuanX Operation Manual, Current Revision

Harding, Soil Analysis Application Report, Low Concentration Soil Contaminant Characterization
Using EDXRF Analysis

Method 6200: Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment

4.0 DEFINITIONS

EDXRF:

Energy Dispersive X-ray Fluorescence Spectrometer/Spectrometry

NIST:

National Institute of Standards and Technology

ppm:

Parts per million

SRM:

Standard Reference Material

QuanX:

Spectrace Brand Name for 6000 Series EDXRF

5.0 RESPONSIBILITIES



REVISION: 0

PAGE:3 OF 11

SOP: XRF INSTRUMENT OPERATION

The **Field Analyst/XRF Operator** is responsible for operation and maintenance of the QuanX EDXRF and data management in accordance with this procedure.

The **Field Supervisor** is responsible for training the Field Analyst(s) on the requirements of this procedure, and for providing oversight and assistance as necessary to ensure compliance with this procedure.

The **Project Chemist** is responsible for reviewing instrument output and quality control records to confirm proper implementation of the quality control requirements.

The Site Health and Safety Officer is responsible for verifying the use of safe laboratory practices throughout implementation of this procedure.

The **Site Manager** is responsible for ensuring that the Field Analyst receives proper training and that proper equipment and resources are available as needed to safely operate the instrument and to generate high quality data.

6.0 EQUIPMENT

- QuanX EDXRF, ECD Model, equipped with spectrometer, control/pulse processing electronics, and data processor
- 20-position sample tray
- CFR 1500 Uninterrupted Power Supply, 100-120/200-240 VAC, 5/2.5 A, 50/60 Hz, single phase, 500 watts (1000 watt total power source to support data processor)
- 3000 LE Series printer

7.0 REQUIREMENTS

7.1 General

A specific area within the field office will be designated as laboratory space for sample preparation and sample analysis using EDXRF. The QuanX must be equipped with an



REVISION: 0

PAGE:4 OF 11

SOP: XRF INSTRUMENT OPERATION

uninterrupted power source, which would provide protection against power surges and allow continuous operation in the event of a power failure.



REVISION: 0

PAGE:5 OF 11

SOP: XRF INSTRUMENT OPERATION

7.2 Maintenance

Maintenance will be performed as specified in the Operations Manual, including:

- Daily Energy Calibration and Calibration Verification
- Weekly Fast Discriminator Check, Log System Status Values
- Semi-annual Interlock Test and Radiation Survey

Any maintenance performed on the instrumentation will be documented in a maintenance log. Additionally, instrument cleaning will be performed using a hand held canister of compressed air to clear dust from the instrument and data processor, in addition to wiping with a clean cloth.

7.3 Instrument Log

A hardbound instrument log book will be used to record the daily instrument operations. The instrument log will be maintained in accordance with the requirements of the Technical Standard Operating Procedure for Field Documentation.

7.4 Quality Control Procedures

- 7.4.1 <u>Date Check</u> Check the system date each day before use by typing *c:\quanx\confirm*> <u>DATE</u>. If the system date displayed in incorrect, type the correct date using the <u>MM-DD-YY</u> format.
- 7.4.2 Energy Calibration The energy calibration is a software function that calibrates the spectrometer electronics to assure the accuracy of the energy scale. An energy calibration will be performed daily prior to use in accordance with the operations manual (Attachment A). If an error message appears, exit the program by pressing the F10 key. If the error reads "ACQ Aborted: Stabilizer Lost Zero Peak," upon exiting it will ask whether to "Save Value of Gain DAC." Type N for NO. Within the Procedure Menu, follow these steps:
 - 1. Press the <u>F7</u> key again to start the energy calibration, pause for approximately 20 seconds and press the space bar to start the program. Repeat this procedure if no response is received.
 - 2. If the first procedure does not work, exit the program by pressing the <u>F10</u> key. Type c:\quanx\confirm> cd and press <u>Enter</u>, repeat to obtain the c: prompt. Turn the unit



REVISION: 0

PAGE:6 OF 11

SOP: XRF INSTRUMENT OPERATION

off, then on and wait approximately one minute. Type <u>cd quanx</u> press <u>Enter</u>, then <u>cd confirm</u> and press <u>Enter</u>. Type <u>Quanx</u> to reach the Procedure Menu. Press the <u>F7</u> key and pause, then press the space bar to start the program.

- 7.4.3 <u>Initial Calibration Verification</u> MK will utilize a Fundamental Parameter method to check the instrument calibration using the following three standard reference materials (SRM), or equivalent:
 - SRM 2709 San Joaquin Soil (Baseline Trace Element Concentrations)
 - SRM 2710 Montana Soil (Highly Elevated Trace Element Concentrations)
 - SRM 2711 Montana Soil (Moderately Elevated Trace Element Concentrations)

Only SRM that have been certified by the National Institute of Standard and Technology shall be utilized, and all certifications shall be reviewed and maintained. The initial calibration verification check will be performed by analyzing the three standards each day, simultaneously, following the energy calibration and prior to analysis of environmental samples. Results will be compared against the NIST acceptable concentration ranges. If one or more concentrations fall outside of the acceptable concentration ranges, the SRM will be re-analyzed. If the results remain outside of the acceptable concentration ranges following three runs, the Troubleshooting Procedures, including a standardization test if necessary, will be performed as specified in the Operations Manual. Prior to proceeding to the standardization test, the following procedures may be performed:

- Turn the sample cup with mylar facing up and tap the container to loosen the soil from the mylar. Shake the sample from side to side and turn back over with mylar facing down, tap on paper towel and place in tray for re-analysis; or
- Replace the mylar.

Environmental samples will not be analyzed without verifying acceptable instrument calibration.

7.4.4 <u>Standardization Test</u> - If the calibration cannot be achieved following the Troubleshooting Procedures, the instrument will be re-standardized. From the Procedure Menu, select "SOIL STANDARDS 6+7". Press the <u>F1</u> key and the program will prompt you to place each SRM in a designated position. Press the space bar to start the program. The printout from



REVISION: 0

PAGE:7 OF 11

SOP: XRF INSTRUMENT OPERATION

this test will be retained and noted in the instrument log; however, the standardization test results will need to be removed from the downloaded electronic data.

- 7.4.5 <u>Continuing Calibration Verification</u> One SRM will be included with the environmental samples for every sample run. The SRM result will be verified against the NIST acceptable concentration range between each run. If the result falls outside of the acceptable range, troubleshooting will be performed as described in 7.4.3 and 7.4.4, and the samples analyzed with that standard will be re-analyzed.
- 7.4.6 <u>Blank Sample</u> Method blanks will be prepared and analyzed using clean silica sand or other material specified in Method 6200. A method blank will be run at least daily to monitor for contamination that may have been introduced through the sample preparation. The method blank is subject to the same preparation procedures as the environmental samples. Detections in the method blank will require that the problem be identified and corrected. Samples analyzed with a blank containing detected elements must be reanalyzed.
- 7.4.7 <u>Performance Evaluation Samples</u> Performance evaluation samples may be analyzed to assess system performance. All performance evaluation samples will be identified and packaged in the same manner as environmental samples and submitted blind to the analyst.
- 7.4.8 <u>Detection Limits</u> The AR Harding Soil Analysis Application Report (attached) presents the lower levels of detection applicable to the QuanX EDXRF (Spectrace 6000) equal to 12 ppm for arsenic and 7 ppm for lead. A detection limit study will be performed as described in Method 6200 to confirm instrument-specific sensitivity on site-specific soil. Ten replicate analysis will be performed on a low-concentration site-specific soil sample with arsenic and lead concentration between 2 and 5 times the expected method detection limit (As: 24 60 ppm, Pb: 14 35 ppm). The method detection limit will be defined as 3 times the standard deviation of the replicate analysis results.

7.5 Sample Analysis Procedures



REVISION: 0

PAGE:8 OF 11

SOP: XRF INSTRUMENT OPERATION

- 7.5.1 Inspect prepared samples to verify condition and proper identification.
- 7.5.2 Enter sample identification into XRF log book (maximum 19 samples per run), and enter the check standard identifications used for that run into log book.
- 7.5.3 Open QuanX cover and load samples into sample tray, matching the log book sequence to the tray numbers. To load samples, tap each sample mylar facing down on a clean paper towel. Place sample in tray with mylar facing down so that the sample sits level. Re-check that log book sequence number matches tray number for each sample.
- 7.5.4 Close QuanX cover.
- 7.5.5 From the Procedure Menu screen select "SOIL UNKNOWNS 2+3".
- 7.5.6 Press the <u>F1</u> key to prompt "HOW MANY SAMPLES TO RUN?" Type in total number (including check standard and method blank if present) from log book and hit enter. From the sample identification screen, enter identifications from log book and press <u>Enter</u> after each entry. Prior to pressing <u>Enter</u> following the last sample identification, re-check the sample identification entries against the log book.
- 7.5.7 Press the final Enter, and the instrument will automatically start the sample analysis.
- 7.5.8 If an error is discovered, cancel this program by pressing the <u>F10</u> key, make the necessary corrections and repeat the procedure.
- 7.5.9 Upon completion of analysis, the QuanX will automatically begin to print the results. The printout will report the elements analyzed, concentration in ppm, and error for each sample identification. Record the concentration of arsenic and lead results in the instrument log book and review quality control sample results as discussed in Section 7.6.
- 7.6 Data Export



NUMBER:

REVISION: 0 PAGE:9 OF 11

SOP: XRF INSTRUMENT OPERATION

The data will be exported at least daily and converted to the project-required format. A daily backup of all data will be maintained. Analytical results will be transferred to the GIS Specialist weekly or more often.



REVISION: 0

PAGE:10 OF 11

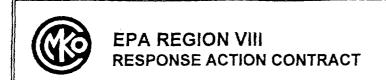
SOP: XRF INSTRUMENT OPERATION

8.0 RECORDS

- NIST Certificates of Analysis for Each Standard Reference Material
- Instrument Log Book
- Maintenance Log
- Instrument Printout

9.0 ATTACHMENTS

- A Spectrace Instruments QuanX Operation Manual
- B Method 6200
- C Soil Analysis Application Report: Low Concentration Soil Contaminant Characterization Using EDXRF Analysis



REVISION: 0

PAGE:11 OF 11

SOP: XRF INSTRUMENT OPERATION

ATTACHMENT A

Spectrace Instruments QuanX Operation Manual

(to be provided by supplier)



REVISION: 0

PAGE:12 OF 11

SOP: XRF INSTRUMENT OPERATION

ATTACHMENT B

Method 6200



REVISION: 0

PAGE:13 OF 11

SOP: XRF INSTRUMENT OPERATION

ATTACHMENT C

Soil Analysis Application Report

Low Concentration Soil Contaminant Characterization Using EDXRF Analysis

ATTACHMENT A

Energy Calibration

Energy Calibration is a software function that calibrates the spectrometer electronics to assure the accuracy of the energy scale. This function requires use of a copper calibration standard supplied with the system.

When run, the program acquires a spectrum of the copper calibration standard and measures the centroid of the Cu K-alpha line. This measured position is compared to the known energy of the Cu line and the difference is displayed as the ERROR. If the error is greater than 1.5eV, the program will adjust the GAIN value and restart. If the error is less than 1.5 eV, the program saves the GAIN value and stops.

It is most important that Energy Calibration be performed on a regular basis. One factor that can affect calibration is temperature, so Energy Calibration should be run at least once a day, unless wide temperature variations are expected, in which case it should be run more often.

- If the program is already running, press EXIT F10, until the Procedure menu is reached. If the program is not running, change to the desired subdirectory and type QUANX.
- 2. To initiate Energy Calibration, press the ENRGY CALIB F7 function key.
- 3. At the prompt load the copper calibration standard, close the lid, and press the space bar on the keyboard to continue.
- 4. The program will initiate the spectrum acquisition and display. If it does, skip to step 6. If an error occurs due to high deadtime, or the acquisition fails to initiate, it will be necessary to change the excitation conditions for Energy calibration before proceeding.
- 5. To change the conditions used for Energy Calibration, press SETUP F2, then SYS STAT F4, then ECAL CONFG F7 to display the Energy Calibration configuration menu. Set to the default conditions: Pd 0.05mm filter 15KV and 0.08mA. Exit to the Procedure menu, then run Energy Calibration.
- 6. With the spectrum acquisition displayed, check that the % Deadtime is near 50%. If it is not, press ACQU MENU F7 then ACQU PARM F4 to display the Acquisition Parameters menu. Now adjust the TUBE CURRENT to achieve 50% deadtime.

 When this is done, press EXIT f10 to return to the acquisition display. Increase voltage by 0.02 mA
- 7. The program will display the spectrum of copper. At the bottom of the screen the GAIN setting and ERROR in the copper peak position are shown. When the program has adjusted the gain, and the error is less than 1.5eV, the system will save the DAC value, and return to the Procedure menu.

Things To Check On a Daily Basis

- 1. On every Monday morning after converting data from the QuanX check the system date. To check the date at the command prompt (C:\QUANX\CONFIRM>) type in the word date and hit enter. This will bring up the current system date. Type in date following this format for example: 07-06-1998 and hit enter. Current date is now entered. Spectrace is aware of this system date problem it is a software error that is caused by running the Redate program.
- 2. Close out previous day runs in the log book. Make sure pages are dated, Pb conc. is recorded, time is entered from the print out, and everything looks good. Make any comments in log book if problems of any runs and why there was a rerun. Make note of any typing mistakes when data is converted the person downloading the data can fix the error.
- 3. Energy Calibration must be ran daily. Place Cu in position #20. At the procedure menu hit F7. The program will prompt you to have Cu sample in position #20 and hit the space bar to start program. The program will go through a 20 second warm-up and then start. Check DT% and make sure it is around 50%. If not follow attachment procedure 5-4. After the warm-up it is important to make sure that there is no errors. The most common one is a message will appear above the spectra saying: ACQ Aborted: Stabilizer Lost Zero Peak. When a error message has appeared exit program by hitting F10. It will ask to save value of Gain DAC. Type N for no. You do not want to save this value. After you type N you will automatically be taken back to the procedure menu. There are two things I have found to have worked with this error:
 - 1. Hit the F7 key again to start energy calibration. Pause for about 20 seconds then hit the space bar to start program. I do not know why this works, but I figure the machine gets tined and needs time to catch up. Try this a couple of times then move to #2.
 - 2. If #1 doesn't work back completely out of the program by hitting F10 to exit. At the C:\Quanx\Confirm>type cd.. And hit enter then type cd.. again and hit enter. Now you only have the c:\ prompt. Behind the Quanx right above the plug in there is a switch to turn the instrument off. Turn it off and wait about one minute. Then type in your directories cd quanx and enter then cd confirm then enter. Now the screen will read

C:\Quanx\Confirm>. Type Quanx and you will be back to the procedure menu. Hit F7 and pause then hit space bar to start program.

- 4. Check standards must run daily after the energy calibration. You only get three chances then it is time to standardize. SRM2711 is the check standard that seems to give me the greatest grief. Couple things to initially try to avoid re-standardization.
 - 1. Turn cup Mylar up and knock soil off of Mylar. Shake side to side and turn back over and tap on paper towel. Place in position #3.
 - 2. If the standard still fails change out the Mylar. There is always a box of Mylar and spare cups behind the pending results tray.
- 5. If you need to standardize at the procedure menu highlight #5 which reads Soil Standards 6+7. Hit F1 and the program will prompt you to place SRM2710 in position #1, SRM2709 in position #2 and SRM2711 in position #3. Hit the space bar and program will start. In the morning when downloading data this information will need to be removed from the worksheet because there is no data to record and will not follow worksheet format.

PROTOCOL FOR ANALYZING SAMPLES ON THE QUANX

- 1. Receive samples from prep lab.
- 2. Look over samples to make sure the writing on the cups is accurate (especially when running assessment samples.)
- 3. Enter sample identifications into log book (19 samples max.)
- 4. Enter check standard identification into log book (next in succession.)
- 5. Check standard must be ran with every batch.
- 6. Open lid to QuanX and load samples according to succession in log book to tray number.

To load samples tap on paper towel MYLAR down. Place in tray MYLAR down and make sure sample is sitting even. Check while loading that number in book is the same for the tray number in the QuanX.

- 7. Close lid to QuanX.
- 8. From the procedure menu screen make sure cursor is highlighting SOIL UNKNOWNS 2+3.
- 9. Hit F1 key to prompt How many samples to run? type in amount from log book and hit enter. That will bring you to entering to sample identifications enter identifications from log book and hit enter after each one. Make sure sample number matches that in the log book.
- 10. Once you have hit enter on the last sample the instrument will automatically start to run.
- 11. If a mistake was made and you need to cancel this program hit the F10 key to exit. Start again at protocol #8.

ATTACHMENT B

- 19. Go to File menu and scroll to Get External Data, then another menu will open and highlight Import.
- 20. Look in drive A. File type is Excel worksheet. Open data file, for ex: 0117.
- 21. A import wizard file will appear. Check the box on the top left for using first row as headers. Hit continue or next at the bottom.
- 22. Now it will ask where do you want to import this data. Select existing table. Scroll down and click on RESULTS. Hit continue or next on bottom.
- 23. This next screen is informing you what table your data will be imported to. Hit finish.
- 24. Your data is now successfully imported to the database.

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT

1.0 SCOPE AND APPLICATION

- 1.1 This method is applicable to the in situ and intrusive analysis of the 26 analytes listed in Table 1 for soil and sediment samples. Some common elements are not listed in Table 1 because they are considered "light" elements that cannot be detected by field portable x-ray fluorescence (FPXRF). They are: lithium, beryllium, sodium, magnesium, aluminum, silicon, and phosphorus. Most of the analytes listed in Table 1 are of environmental concern, while a few others have interference effects or change the elemental composition of the matrix, affecting quantitation of the analytes of interest. Generally elements of atomic number 16 or greater can be detected and quantitated by FPXRF.
- 1.2 Detection limits depend on several factors, the analyte of interest, the type of detector used, the type of excitation source, the strength of the excitation source, count times used to irradiate the sample, physical matrix effects, chemical matrix effects, and interelement spectral interferences. General instrument detection limits for analytes of interest in environmental applications are shown in Table 1. These detection limits apply to a clean matrix of quartz sand (silicon dioxide) free of interelement spectral interferences using long (600-second) count times. These detection limits are given for guidance only and will vary depending on the sample matrix, which instrument is used, and operating conditions. A discussion of field performance-based detection limits is presented in Section 13.4 of this method. The clean matrix and field performance-based detection limits should be used for general planning purposes, and a third detection limit discussed, based on the standard deviation around single measurements, should be used in assessing data quality. This detection limit is discussed in Sections 9.7 and 11.3.
- 1.3 Use of this method is restricted to personnel either trained and knowledgeable in the operation of an XRF instrument or under the supervision of a trained and knowledgeable individual. This method is a screening method to be used with confirmatory analysis using EPA-approved methods. This method's main strength is as a rapid field screening procedure. The method detection limits (MDL) of FPXRF are above the toxicity characteristic regulatory level for most RCRA analytes. If the precision, accuracy, and detection limits of FPXRF meet the data quality objectives (DQOs) of your project, then XRF is a fast, powerful, cost effective technology for site characterization.

2.0 SUMMARY OF METHOD

2.1 The FPXRF technologies described in this method use sealed radioisotope sources to irradiate samples with x-rays. X-ray tubes are used to irradiate samples in the laboratory and are beginning to be incorporated into field portable instruments. When a sample is irradiated with x-rays, the source x-rays may undergo either scattering or absorption by sample atoms. This later process is known as the photoelectric effect. When an atom absorbs the source x-rays, the incident radiation dislodges electrons from the innermost shells of the atom, creating vacancies. The electron vacancies are filled by electrons cascading in from outer electron shells. Electrons in outer shells have higher energy states than inner shell electrons, and the outer shell electrons give off energy as they cascade down into the inner shell vacancies. This rearrangement of electrons results in emission of x-rays characteristic of the given atom. The emission of x-rays, in this manner, is termed x-ray fluorescence.

proportional to the energy of the x-rays. An electronic multichannel analyzer (MCA) measures the pulse amplitudes, which is the basis of qualitative x-ray analysis. The number of counts at a given energy per unit of time is representative of the element concentration in a sample and is the basis for quantitative analysis. Most FPXRF instruments are menu-driven from software built into the units or from personal computers (PC).

The measurement time of each source is user-selectable. Shorter source measurement times (30 seconds) are generally used for initial screening and hot spot delineation, and longer measurement times (up to 300 seconds) are typically used to meet higher precision and accuracy requirements.

FPXRF instruments can be calibrated using the following methods: internally using fundamental parameters determined by the manufacturer, empirically based on site-specific calibration standards (SSCS), or based on Compton peak ratios. The Compton peak is produced by backscattering of the source radiation. Some FPXRF instruments can be calibrated using multiple methods.

3.0 DEFINITIONS

- 3.1 <u>FPXRF</u>: Field portable x-ray fluorescence.
- 3.2 MCA: Multichannel analyzer for measuring pulse amplitude.
- 3.3 <u>SSCS</u>: Site specific calibration standard.
- 3.4 <u>FP</u>: Fundamental parameter.
- 3.5 ROI: Region of interest.
- 3.6 <u>SRM</u>: Standard reference material. A standard containing certified amounts of metals in soil or sediment.
- 3.7 <u>eV</u>: Electron Volt. A unit of energy equivalent to the amount of energy gained by an electron passing through a potential difference of one volt.
 - 3.8 Refer to Chapter One and Chapter Three for additional definitions.

4.0 INTERFERENCES

- 4.1 The total method error for FPXRF analysis is defined as the square root of the sum of squares of both instrument precision and user- or application-related error. Generally, instrument precision is the least significant source of error in FPXRF analysis. User- or application-related error is generally more significant and varies with each site and method used. Some sources of interference can be minimized or controlled by the instrument operator, but others cannot. Common sources of user- or application-related error are discussed below.
- 4.2 Physical matrix effects result from variations in the physical character of the sample. These variations may include such parameters as particle size, uniformity, homogeneity, and surface condition. For example, if any analyte exists in the form of very fine particles in a coarser-grained matrix, the analyte's concentration measured by the FPXRF will vary depending on how fine particles are distributed within the coarser-grained matrix. If the fine particles "settle" to the bottom of the sample cup, the analyte concentration measurement will be higher than if the fine particles are not

mixed in well and stay on top of the coarser-grained particles in the sample cup. One way to reduce such error is to grind and sieve all soil samples to a uniform particle size thus reducing sample-to-sample particle size variability. Homogeneity is always a concern when dealing with soil samples. Every effort should be made to thoroughly mix and homogenize soil samples before analysis. Field studies have shown heterogeneity of the sample generally has the largest impact on comparability with confirmatory samples.

4.3 Moisture content may affect the accuracy of analysis of soil and sediment sample analyses. When the moisture content is between 5 and 20 percent, the overall error from moisture may be minimal. However, moisture content may be a major source of error when analyzing samples of surface soil or sediment that are saturated with water. This error can be minimized by drying the samples in a convection or toaster oven. Microwave drying is not recommended because field studies have shown that microwave drying can increase variability between FPXRF data and confirmatory analysis and because metal fragments in the sample can cause arcing to occur in a microwave.

| PRCP. | 16.1.3

- 4.4 Inconsistent positioning of samples in front of the probe window is a potential source of error because the x-ray signal decreases as the distance from the radioactive source increases. This error is minimized by maintaining the same distance between the window and each sample. For the best results, the window of the probe should be in direct contact with the sample, which means that the sample should be flat and smooth to provide a good contact surface.
- 4.5 Chemical matrix effects result from differences in the concentrations of interfering elements. These effects occur as either spectral interferences (peak overlaps) or as x-ray absorption and enhancement phenomena. Both effects are common in soils contaminated with heavy metals. As examples of absorption and enhancement effects; iron (Fe) tends to absorb copper (Cu) x-rays, reducing the intensity of the Cu measured by the detector, while chromium (Cr) will be enhanced at the expense of Fe because the absorption edge of Cr is slightly lower in energy than the fluorescent peak of iron. The effects can be corrected mathematically through the use of fundamental parameter (FP) coefficients. The effects also can be compensated for using SSCS, which contain all the elements present on site that can interfere with one another.
- 4.6 When present in a sample, certain x-ray lines from different elements can be very close in energy and, therefore, can cause interference by producing a severely overlapped spectrum. The degree to which a detector can resolve the two different peaks depends on the energy resolution of the detector. If the energy difference between the two peaks in electron volts is less than the resolution of the detector in electron volts, then the detector will not be able to fully resolve the peaks.

The most common spectrum overlaps involve the K_{β} line of element Z-1 with the K_{α} line of element Z. This is called the K_{α}/K_{β} interference. Because the $K_{\alpha}:K_{\beta}$ intensity ratio for a given element usually is about 7:1, the interfering element, Z-1, must be present at large concentrations to cause a problem. Two examples of this type of spectral interference involve the presence of large concentrations of vanadium (V) when attempting to measure Cr or the presence of large concentrations of Fe when attempting to measure cobalt (Co). The V K_{α} and K_{β} energies are 4.95 and 5.43 keV, respectively, and the Cr K_{α} energy is 5.41 keV. The Fe K_{α} and K_{β} energies are 6.40 and 7.06 keV, respectively, and the Co K_{α} energy is 6.92 keV. The difference between the V K_{β} and Cr K_{α} energies is 20 eV, and the difference between the Fe K_{β} and the Co K_{α} energies is 140 eV. The resolution of the highest-resolution detectors in FPXRF instruments is 170 eV. Therefore, large amounts of V and Fe will interfere with quantitation of Cr or Co, respectively. The presence of Fe is a frequent problem because it is often found in soils at tens of thousands of parts per million (ppm).

4.7 Other interferences can arise from K/L, K/M, and L/M line overlaps, although these overlaps are less common. Examples of such overlap involve arsenic (As) K_{α} /lead (Pb) L_{α} and sulfur (S) K_{α} /Pb M_{α} . In the As/Pb case, Pb can be measured from the Pb L_{β} line, and As can be measured from either the As K_{α} or the As K_{α} line; in this way the interference can be corrected. If the As K_{β} line is used, sensitivity will be decreased by a factor of two to five times because it is a less intense line than the As K_{α} line. If the As K_{α} line is used in the presence of Pb, mathematical corrections within the instrument software can be used to subtract out the Pb interference. However, because of the limits of mathematical corrections, As concentrations cannot be efficiently calculated for samples with Pb:As ratios of 10:1 or more. This high ratio of Pb to As may result in no As being reported regardless of the actual concentration present.

No instrument can fully compensate for this interference. It is important for an operator to understand this limitation of FPXRF instruments and consult with the manufacturer of the FPXRF instrument to evaluate options to minimize this limitation. The operator's decision will be based on action levels for metals in soil established for the site, matrix effects, capabilities of the instrument, data quality objectives, and the ratio of lead to arsenic known to be present at the site. If a site is encountered that contains lead at concentrations greater than ten times the concentration of arsenic it is advisable that all critical soil samples be sent off site for confirmatory analysis by an EPA-approved method.

- 4.8 If SSCS are used to calibrate an FPXRF instrument, the samples collected must be representative of the site under investigation. Representative soil sampling ensures that a sample or group of samples accurately reflects the concentrations of the contaminants of concern at a given time and location. Analytical results for representative samples reflect variations in the presence and concentration ranges of contaminants throughout a site. Variables affecting sample representativeness include differences in soil type, contaminant concentration variability, sample collection and preparation variability, and analytical variability, all of which should be minimized as much as possible.
- 4.9 Soil physical and chemical effects may be corrected using SSCS that have been analyzed by inductively coupled plasma (ICP) or atomic absorption (AA) methods. However, a major source of error can be introduced if these samples are not representative of the site or if the analytical error is large. Another concern is the type of digestion procedure used to prepare the soil samples for the reference analysis. Analytical results for the confirmatory method will vary depending on whether a partial digestion procedure, such as SW-846 Method 3050, or a total digestion procedure, such as Method 3052 is used. It is known that depending on the nature of the soil or sediment, Method 3050 will achieve differing extraction efficiencies for different analytes of interest. The confirmatory method should meet the project data quality objectives.

XRF measures the total concentration of an element; therefore, to achieve the greatest comparability of this method with the reference method (reduced bias), a total digestion procedure should be used for sample preparation. However, in the study used to generate the performance data for this method, the confirmatory method used was Method 3050, and the FPXRF data compared very well with regression correlation coefficients (r² often exceeding 0.95, except for barium and chromium. See Table 9 in Section 17.0). The critical factor is that the digestion procedure and analytical reference method used should meet the data quality objectives (DQOs) of the project and match the method used for confirmation analysis.

4.10 Ambient temperature changes can affect the gain of the amplifiers producing instrument drift. Gain or drift is primarily a function of the electronics (amplifier or preamplifier) and not the detector as most instrument detectors are cooled to a constant temperature. Most FPXRF instruments have a built-in automatic gain control. If the automatic gain control is allowed to make

periodic adjustments, the instrument will compensate for the influence of temperature changes on its energy scale. If the FPXRF instrument has an automatic gain control function, the operator will not have to adjust the instrument's gain unless an error message appears. If an error message appears, the operator should follow the manufacturer's procedures for troubleshooting the problem. Often, this involves performing a new energy calibration. The performance of an energy calibration check to assess drift is a quality control measure discussed in Section 9.2.

If the operator is instructed by the manufacturer to manually conduct a gain check because of increasing or decreasing ambient temperature, it is standard to perform a gain check after every 10 to 20 sample measurements or once an hour whichever is more frequent. It is also suggested that a gain check be performed if the temperature fluctuates more than 10 to 20°F. The operator should follow the manufacturer's recommendations for gain check frequency.

5.0 SAFETY

- 5.1 Proper training for the safe operation of the instrument and radiation training should be completed by the analyst prior to analysis. Radiation safety for each specific instrument can be found in the operators manual. Protective shielding should never be removed by the analyst or any personnel other than the manufacturer. The analyst should be aware of the local state and national regulations that pertain to the use of radiation-producing equipment and radioactive materials with which compliance is required. Licenses for radioactive materials are of two types; (1) general license which is usually provided by the manufacturer for receiving, acquiring, owning, possessing, using, and transferring radioactive material incorporated in a device or equipment, and (2) specific license which is issued to named persons for the operation of radioactive instruments as required by local state agencies. There should be a person appointed within the organization that is solely responsible for properly instructing all personnel, maintaining inspection records, and monitoring xray equipment at regular intervals. A copy of the radioactive material licenses and leak tests should be present with the instrument at all times and available to local and national authorities upon request. X-ray tubes do not require radioactive material licenses or leak tests, but do require approvals and licenses which vary from state to state. In addition, fail-safe x-ray warning lights should be illuminated whenever an x-ray tube is energized. Provisions listed above concerning radiation safety regulations, shielding, training, and responsible personnel apply to x-ray tubes just as to radioactive sources. In addition, a log of the times and operating conditions should be kept whenever an x-ray tube is energized. Finally, an additional hazard present with x-ray tubes is the danger of electric shock from the high voltage supply. The danger of electric shock is as substantial as the danger from radiation but is often overlooked because of its familiarity.
- 5.2 Radiation monitoring equipment should be used with the handling of the instrument. The operator and the surrounding environment should be monitored continually for analyst exposure to radiation. Thermal luminescent detectors (TLD) in the form of badges and rings are used to monitor operator radiation exposure. The TLDs should be worn in the area of most frequent exposure. The maximum permissible whole-body dose from occupational exposure is 5 Roentgen Equivalent Man (REM) per year. Possible exposure pathways for radiation to enter the body are ingestion, inhaling, and absorption. The best precaution to prevent radiation exposure is distance and shielding.
 - 5.3 Refer to Chapter Three for guidance on some proper safety protocols.

6.0 EQUIPMENT AND SUPPLIES

6.1 <u>FPXRF Spectrometer</u>: An FPXRF spectrometer consists of four major components: (1) a source that provides x-rays; (2) a sample presentation device; (3) a detector that converts x-

ray-generated photons emitted from the sample into measurable electronic signals; and (4) a data processing unit that contains an emission or fluorescence energy analyzer, such as an MCA, that processes the signals into an x-ray energy spectrum from which elemental concentrations in the sample may be calculated, and a data display and storage system. These components and additional, optional items, are discussed below.

6.1.1 Excitation Sources: Most FPXRF instruments use sealed radioisotope sources to produce x-rays in order to irradiate samples. The FPXRF instrument may contain between one and three radioisotope sources. Common radioisotope sources used for analysis for metals in soils are iron (Fe)-55, cadmium (Cd)-109, americium (Am)-241, and curium (Cm)-244. These sources may be contained in a probe along with a window and the detector; the probe is connected to a data reduction and handling system by means of a flexible cable. Alternatively, the sources, window, and detector may be included in the same unit as the data reduction and handling system.

The relative strength of the radioisotope sources is measured in units of millicuries (mCi). All other components of the FPXRF system being equal, the stronger the source, the greater the sensitivity and precision of a given instrument. Radioisotope sources undergo constant decay. In fact, it is this decay process that emits the primary x-rays used to excite samples for FPXRF analysis. The decay of radioisotopes is measured in "half-lives." The half-life of a radioisotope is defined as the length of time required to reduce the radioisotopes strength or activity by half. Developers of FPXRF technologies recommend source replacement at regular intervals based on the source's half-life. The characteristic x-rays emitted from each of the different sources have energies capable of exciting a certain range of analytes in a sample. Table 2 summarizes the characteristics of four common radioisotope sources.

X-ray tubes have higher radiation output, no intrinsic lifetime limit, produce constant output over their lifetime, and do not have the disposal problems of radioactive sources but are just now appearing in FPXRF instruments. An electrically-excited x-ray tube operates by bombarding an anode with electrons accelerated by a high voltage. The electrons gain an energy in electron volts equal to the accelerating voltage and can excite atomic transitions in the anode, which then produces characteristic x-rays. These characteristic x-rays are emitted through a window which contains the vacuum required for the electron acceleration. An important difference between x-ray tubes and radioactive sources is that the electrons which bombard the anode also produce a continuum of x-rays across a broad range of energies in addition to the characteristic x-rays. This continuum is weak compared to the characteristic x-rays but can provide substantial excitation since it covers a broad energy range. It has the undesired property of producing background in the spectrum near the analyte x-ray lines when it is scattered by the sample. For this reason a filter is often used between the x-ray tube and the sample to suppress the continuum radiation while passing the characteristic x-rays from the anode. This filter is sometimes incorporated into the window of the x-ray tube. The choice of accelerating voltage is governed by the anode material, since the electrons must have sufficient energy to excite the anode, which requires a voltage greater than the absorption edge of the anode material. The anode is most efficiently excited by voltages 2 to 2.5 times the edge energy (most x-rays per unit power to the tube), although voltages as low as 1.5 times the absorption edge energy will work. The characteristic x-rays emitted by the anode are capable of exciting a range of elements in the sample just as with a radioactive source. Table 3 gives the recommended operating voltages and the sample elements excited for some common anodes.

- 6.1.2 Sample Presentation Device: FPXRF instruments can be operated in two modes: in situ and intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. For most FPXRF instruments operated in the intrusive mode, the probe is rotated so that the window faces upward. A protective sample cover is placed over the window, and the sample cup is placed on top of the window inside the protective sample cover for analysis.
- 6.1.3 Detectors: The detectors in the FPXRF instruments can be either solid-state detectors or gas-filled, proportional counter detectors. Common solid-state detectors include mercuric iodide (Hgl_2), silicon pin diode and lithium-drifted silicon Si(Li). The Hgl_2 detector is operated at a moderately subambient temperature controlled by a low power thermoelectric cooler. The silicon pin diode detector also is cooled via the thermoelectric Peltier effect. The Si(Li) detector must be cooled to at least -90 °C either with liquid nitrogen or by thermoelectric cooling via the Peltier effect. Instruments with a Si(Li) detector have an internal liquid nitrogen dewar with a capacity of 0.5 to 1.0 liter. Proportional counter detectors are rugged and lightweight, which are important features of a field portable detector. However, the resolution of a proportional counter detector is not as good as that of a solid-state detector. The energy resolution of a detector for characteristic x-rays is usually expressed in terms of full width at half-maximum (FWHM) height of the manganese K_{α} peak at 5.89 keV. The typical resolutions of the above mentioned detectors are as follows: Hgl_2 -270 eV; silicon pin diode-250 eV; Si(Li)-170 eV; and gas-filled, proportional counter-750 eV.

During operation of a solid-state detector, an x-ray photon strikes a biased, solid-state crystal and loses energy in the crystal by producing electron-hole pairs. The electric charge produced is collected and provides a current pulse that is directly proportional to the energy of the x-ray photon absorbed by the crystal of the detector. A gas-filled, proportional counter detector is an ionization chamber filled with a mixture of noble and other gases. An x-ray photon entering the chamber ionizes the gas atoms. The electric charge produced is collected and provides an electric signal that is directly proportional to the energy of the x-ray photon absorbed by the gas in the detector.

- 6.1.4 Data Processing Units: The key component in the data processing unit of an FPXRF instrument is the MCA. The MCA receives pulses from the detector and sorts them by their amptitudes (energy level). The MCA counts pulses per second to determine the height of the peak in a spectrum, which is indicative of the target analyte's concentration. The spectrum of element peaks are built on the MCA. The MCAs in FPXRF instruments have from 256 to 2,048 channels. The concentrations of target analytes are usually shown in parts per million on a liquid crystal display (LCD) in the instrument. FPXRF instruments can store both spectra and from 100 to 500 sets of numerical analytical results. Most FPXRF instruments are menu-driven from software built into the units or from PCs. Once the data—storage memory of an FPXRF unit is full, data can be downloaded by means of an RS-232 port and cable to a PC.
- 6.2 Spare battery chargers.
- 6.3 Polyethylene sample cups: 31 millimeters (mm) to 40 mm in diameter with collar, or equivalent (appropriate for FPXRF instrument).
- 6.4 X-ray window film: Mylar[™], Kapton[™], Spectrolene[™], polypropylene, or equivalent; 2.5 to 6.0 micrometers (µm) thick.

- Mortar and pestle: glass, agate, or aluminum oxide; for grinding soil and sediment the 6.5 samples.
 - 6.6 Containers: glass or plastic to store samples.
- Sieves: 60-mesh (0.25 mm), stainless-steel, Nylon, or equivalent for preparing soil and PRCO 6.7 sediment samples.
 - 6.8 Trowels: for smoothing soil surfaces and collecting soil samples.
 - Plastic bags: used for collection and homogenization of soil samples. (Prep., Collection) 6.9
- Drying oven: standard convection or toaster oven, for soil and sediment samples that Prep. 6.10 require drying.

7.0 REAGENTS AND STANDARDS

- Pure Element Standards: Each pure, single-element standard is intended to produce strong characteristic x-ray peaks of the element of interest only. Other elements present must not contribute to the fluorescence spectrum. A set of pure element standards for commonly sought analytes is supplied by the instrument manufacturer, if required for the instrument; not all instruments require the pure element standards. The standards are used to set the region of interest (ROI) for each element. They also can be used as energy calibration and resolution check samples.
- Site-specific Calibration Standards: Instruments that employ fundamental parameters (FP) or similar mathematical models in minimizing matrix effects may not require SSCS. If the FP calibration model is to be optimized or if empirical calibration is necessary, then SSCSs must be collected, prepared, and analyzed.
 - The SSCS must be representative of the matrix to be analyzed by FPXRF. These samples must be well homogenized. A minimum of ten samples spanning the concentration ranges of the analytes of interest and of the interfering elements must be obtained from the site. A sample size of 4 to 8 ounces is recommended, and standard glass sampling jars should be used.
 - 7.2.2 Each sample should be oven-dried for 2 to 4 hours at a temperature of less than 150°C. If mercury is to be analyzed, a separate sample portion must remain undried, as / γιερ. heating may volatilize the mercury. When the sample is dry, all large, organic debris and nonrepresentative material, such as twigs, leaves, roots, insects, asphalt, and rock should be removed. The sample should be ground with a mortar and pestle and passed through a 60mesh sieve. Only the coarse rock fraction should remain on the screen.
 - The sample should be homogenized by using a riffle splitter or by placing 150 to 200 grams of the dried, sieved sample on a piece of kraft or butcher paper about 1.5 by 1.5 feet in size. Each corner of the paper should be lifted alternately, rolling the soil over on itself and toward the opposite corner. The soil should be rolled on itself 20 times. Approximately 5 grams of the sample should then be removed and placed in a sample cup for FPXRF analysis. The rest of the prepared sample should be sent off site for ICP or AA analysis. The method use for confirmatory analysis should meet the data quality objectives of the project.
- Blank Samples: The blank samples should be from a "clean" guartz or silicon dioxide matrix that is free of any analytes at concentrations above the method detection limits. These

6.3.3

samples are used to monitor for cross-contamination and laboratory-induced contaminants or interferences.

7.4 Standard Reference Materials: Standard reference materials (SRM) are standards containing certified amounts of metals in soil or sediment. These standards are used for accuracy and performance checks of FPXRF analyses. SRMs can be obtained from the National Institute of Standards and Technology (NIST), the U.S. Geological Survey (USGS), the Canadian National Research Council, and the national bureau of standards in foreign nations. Pertinent NIST SRMs for FPXRF analysis include 2704, Buffalo River Sediment; 2709, San Joaquin Soil; and 2710 and 2711, Montana Soil. These SRMs contain soil or sediment from actual sites that has been analyzed using independent inorganic analytical methods by many different laboratories.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

Sample handling and preservation procedures used in FPXRF analyses should follow the guidelines in Chapter Three, Inorganic Analytes.

9.0 QUALITY CONTROL

- 9.1 Refer to Chapter One for additional guidance on quality assurance protocols. All field data sheets and quality control data should be maintained for reference or inspection.
- 9.2 Energy Calibration Check: To determine whether an FPXRF instrument is operating within resolution and stability tolerances, an energy calibration check should be run. The energy calibration check determines whether the characteristic x-ray lines are shifting, which would indicate drift within the instrument. As discussed in Section 4.10, this check also serves as a gain check in the event that ambient temperatures are fluctuating greatly (> 10 to 20°F).

The energy calibration check should be run at a frequency consistent with manufacturers recommendations. Generally, this would be at the beginning of each working day, after the batteries are changed or the instrument is shut off, at the end of each working day, and at any other time when the instrument operator believes that drift is occurring during analysis. A pure element such as iron, manganese, copper, or lead is often used for the energy calibration check. A manufacturer-recommended count time per source should be used for the check.

- 9.2.1 The instrument manufacturer's manual specifies the channel or kiloelectron volt level at which a pure element peak should appear and the expected intensity of the peak. The intensity and channel number of the pure element as measured using the radioactive source should be checked and compared to the manufacturer's recommendation. If the energy calibration check does not meet the manufacturer's criteria, then the pure element sample should be repositioned and reanalyzed. If the criteria are still not met, then an energy calibration should be performed as described in the manufacturer's manual. With some FPXRF instruments, once a spectrum is acquired from the energy calibration check, the peak can be optimized and realigned to the manufacturer's specifications using their software.
- 9.3 Blank Samples: Two types of blank samples should be analyzed for FPXRF analysis: instrument blanks and method blanks. An instrument blank is used to verify that no contamination exists in the spectrometer or on the probe window.
 - 9.3.1 The instrument blank can be silicon dioxide, a Teflon block, a quartz block, "clean" sand, or lithium carbonate. This instrument blank should be analyzed on each working day before and after analyses are conducted and once per every twenty samples. An

instrument blank should also be analyzed whenever contamination is suspected by the analyst. The frequency of analysis will vary with the data quality objectives of the project. A manufacturer-recommended count time per source should be used for the blank analysis. No element concentrations above the method detection limits should be found in the instrument blank. If concentrations exceed these limits, then the probe window and the check sample should be checked for contamination. If contamination is not a problem, then the instrument must be "zeroed" by following the manufacturer's instructions.

- 9.3.2 A method blank is used to monitor for laboratory-induced contaminants or interferences. The method blank can be "clean" silica sand or lithium carbonate that undergoes the same preparation procedure as the samples. A method blank must be analyzed at least daily. The frequency of analysis will depend on the data quality objectives of the project. To be acceptable, a method blank must not contain any analyte at a concentration above its method detection limit. If an analyte's concentration exceeds its method detection limit, the cause of the problem must be identified, and all samples analyzed with the method blank must be reanalyzed.
- 9.4 Calibration Verification Checks: A calibration verification check sample is used to check the accuracy of the instrument and to assess the stability and consistency of the analysis for the analytes of interest. A check sample should be analyzed at the beginning of each working day, during active sample analyses, and at the end of each working day. The frequency of calibration checks during active analysis will depend on the data quality objectives of the project. The check sample should be a well characterized soil sample from the site that is representative of site samples in terms of particle size and degree of homogeneity and that contains contaminants at concentrations near the action levels. If a site-specific sample is not available, then an NIST or other SRM that contains the analytes of interest can be used to verify the accuracy of the instrument. The measured value for each target analyte should be within ±20 percent (%D) of the true value for the calibration verification check to be acceptable. If a measured value falls outside this range, then the check sample should be recalibrated, and the batch of samples analyzed before the unacceptable calibration verification check must be reanalyzed.
- 9.5 Precision Measurements: The precision of the method is monitored by analyzing a sample with low, moderate, or high concentrations of target analytes. The frequency of precision measurements will depend on the data quality objectives for the data. A minimum of one precision sample should be run per day. Each precision sample should be analyzed 7 times in replicate. It is recommended that precision measurements be obtained for samples with varying concentration ranges to assess the effect of concentration on method precision. Determining method precision for analytes at concentrations near the site action levels can be extremely important if the FPXRF results are to be used in an enforcement action; therefore, selection of at least one sample with target analyte concentrations at or near the site action levels or levels of concern is recommended. A precision sample is analyzed by the instrument for the same field analysis time as used for other project samples. The relative standard deviation (RSD) of the sample mean is used to assess method precision. For FPXRF data to be considered adequately precise, the RSD should not be greater than 20 percent with the exception of chromium. RSD values for chromium should not be greater than 30 percent.

The equation for calculating RSD is as follows:

RSD = (SD/Mean Concentration) x 100

where:

RSD = Relative standard deviation for the precision measurement for the

analyte

SD = Standard deviation of the concentration for the analyte

Mean Concentration = Mean concentration for the analyte

The precision or reproducibility of a measurement will improve with increasing count time, however, increasing the count time by a factor of 4 will provide only 2 times better precision, so there is a point of diminishing return. Increasing the count time also improves the detection limit, but decreases sample throughput.

9.6 Detection Limits: Results for replicate analyses of a low-concentration sample, SSCS, or SRM can be used to generate an average site-specific method detection and quantitation limits. In this case, the method detection limit is defined as 3 times the standard deviation of the results for the low-concentration samples and the method quantitation limit is defined as 10 times the standard deviation of the same results. Another means of determining method detection and quantitation limits involves use of counting statistics. In FPXRF analysis, the standard deviation from counting statistics is defined as SD = $(N)^{\frac{1}{2}}$, where SD is the standard deviation for a target analyte peak and N is the net counts for the peak of the analyte of interest (i.e., gross counts minus background under the peak). Three times this standard deviation would be the method detection limit and 10 times this standard deviation would be the method quantitation limit. If both of the above mentioned approaches are used to calculate method detection limits, the larger of the standard deviations should be used to provide the more conservative detection limits.

This SD based detection limit criteria must be used by the operator to evaluate each measurement for its useability. A measurement above the average calculated or manufacturer's detection limit, but smaller than three times its associated SD, should not be used as a quantitative measurement. Conversely, if the measurement is below the average calculated or manufacturer's detection limit, but greater than three times its associated SD. It should be coded as an estimated value.

9.7 Confirmatory Samples: The comparability of the FPXRF analysis is determined by submitting FPXRF-analyzed samples for analysis at a laboratory. The method of confirmatory analysis must meet the project and XRF measurement data quality objectives. The confirmatory samples must be splits of the well homogenized sample material. In some cases the prepared sample cups can be submitted. A minimum of 1 sample for each 20 FPXRF-analyzed samples should be submitted for confirmatory analysis. This frequency will depend on data quality objectives. The confirmatory analyses can also be used to verify the quality of the FPXRF data. The confirmatory samples should be selected from the lower, middle, and upper range of concentrations measured by the FPXRF. They should also include samples with analyte concentrations at or near the site action levels. The results of the confirmatory analysis and FPXRF analyses should be evaluated with a least squares linear regression analysis. If the measured concentrations span more than one order of magnitude, the data should be log-transformed to standardize variance which is proportional to the magnitude of measurement. The correlation coefficient (r^2) for the results should be 0.7 or greater for the FPXRF data to be considered screening level data. If the r² is 0.9 or greater and inferential statistics indicate the FPXRF data and the confirmatory data are statistically equivalent at a 99 percent confidence level, the data could potentially meet definitive level data criteria.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 Instrument Calibration: Instrument calibration procedures vary among FPXRF instruments. Users of this method should follow the calibration procedures outlined in the operator's manual for each specific FPXRF instrument. Generally, however, three types of calibration procedures exist for FPXRF instruments: FP calibration, empirical calibration, and the Compton peak ratio or normalization method. These three types of calibration are discussed below.
- 10.2 Fundamental Parameters Calibration: FP calibration procedures are extremely variable. An FP calibration provides the analyst with a "standardless" calibration. The advantages of FP calibrations over empirical calibrations include the following:
 - No previously collected site-specific samples are required, although site-specific samples with confirmed and validated analytical results for all elements present could be used.
 - Cost is reduced because fewer confirmatory laboratory results or calibration standards are required.

However, the analyst should be aware of the limitations imposed on FP calibration by particle size and matrix effects. These limitations can be minimized by adhering to the preparation procedure described in Section 7.2. The two FP calibration processes discussed below are based on an effective energy FP routine and a back scatter with FP (BFP) routine. Each FPXRF FP calibration process is based on a different iterative algorithmic method. The calibration procedure for each routine is explained in detail in the manufacturer's user manual for each FPXRF instrument; in addition, training courses are offered for each instrument.

10.2.1 Effective Energy FP Calibration: The effective energy FP calibration is performed by the manufacturer before an instrument is sent to the analyst. Although SSCS can be used, the calibration relies on pure element standards or SRMs such as those obtained from NIST for the FP calibration. The effective energy routine relies on the spectrometer response to pure elements and FP iterative algorithms to compensate for various matrix effects.

Alpha coefficients are calculated using a variation of the Sherman equation, which calculates theoretical intensities from the measurement of pure element samples. These coefficients indicate the quantitative effect of each matrix element on an analyte's measured x-ray intensity. Next, the Lachance Traill algorithm is solved as a set of simultaneous equations based on the theoretical intensities. The alpha coefficients are then downloaded into the specific instrument.

The working effective energy FP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of sampling. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. A manufacturer-recommended count time per source should be used for the calibration check. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A percent difference (%D) is then calculated for each target analyte. The %D should be within ±20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the

line or the y-intercept value for the analyte. The SRM or SSCS is reanalyzed until the %D falls within ±20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

The equation to calibrate %D is as follows:

 $%D = ((C_s - C_k) / C_k) \times 100$

where:

%D = Percent difference

 C_k = Certified concentration of standard sample C_s = Measured concentration of standard sample

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10.2.2 BFP Calibration: BFP calibration relies on the ability of the liquid nitrogen-cooled, Si(Li) solid-state detector to separate the coherent (Compton) and incoherent (Rayleigh) backscatter peaks of primary radiation. These peak intensities are known to be a function of sample composition, and the ratio of the Compton to Rayleigh peak is a function of the mass absorption of the sample. The calibration procedure is explained in detail in the instrument manufacturer's manual. Following is a general description of the BFP calibration procedure.

The concentrations of all detected and quantified elements are entered into the computer software system. Certified element results for an NIST SRM or confirmed and validated results for an SSCS can be used. In addition, the concentrations of oxygen and silicon must be entered; these two concentrations are not found in standard metals analyses. The manufacturer provides silicon and oxygen concentrations for typical soil types. Pure element standards are then analyzed using a manufacturer-recommended count time per source. The results are used to calculate correction factors in order to adjust for spectrum overlap of elements.

The working BFP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of the analysis. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. The standard sample is analyzed using a manufacturer-recommended count time per source to check the calibration curve. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A %D is then calculated for each target analyte. The %D should fall within ±20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line the y-intercept value for the analyte. The standard sample is reanalyzed until the %D falls within ±20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

10.3 Empirical Calibration: An empirical calibration can be performed with SSCS, site-typical standards, or standards prepared from metal oxides. A discussion of SSCS is included in Section 7.2; if no previously characterized samples exist for a specific site, site-typical standards can be used. Site-typical standards may be selected from commercially available characterized soils or from SSCS prepared for another site. The site-typical standards should closely approximate the site's soil matrix with respect to particle size distribution, mineralogy, and contaminant analytes. If neither SSCS nor site-typical standards are available, it is possible to make gravimetric standards

by adding metal oxides to a "clean" sand or silicon dioxide matrix that simulates soil. Metal oxides can be purchased from various chemical vendors. If standards are made on site, a balance capable of weighing items to at least two decimal places is required. Concentrated ICP or AA standard solutions can also be used to make standards. These solutions are available in concentrations of 10,000 parts per million, thus only small volumes have to be added to the soil.

An empirical calibration using SSCS involves analysis of SSCS by the FPXRF instrument and by a conventional analytical method such as ICP or AA. A total acid digestion procedure should be used by the laboratory for sample preparation. Generally, a minimum of 10 and a maximum of 30 well characterized SSCS, site-typical standards, or prepared metal oxide standards are required to perform an adequate empirical calibration. The number of required standards depends on the number of analytes of interest and interfering elements. Theoretically, an empirical calibration with SSCS should provide the most accurate data for a site because the calibration compensates for site-specific matrix effects.

The first step in an empirical calibration is to analyze the pure element standards for the elements of interest. This enables the instrument to set channel limits for each element for spectral deconvolution. Next the SSCS, site-typical standards, or prepared metal oxide standards are analyzed using a count time of 200 seconds per source or a count time recommended by the manufacturer. This will produce a spectrum and net intensity of each analyte in each standard. The analyte concentrations for each standard are then entered into the instrument software; these concentrations are those obtained from the laboratory, the certified results, or the gravimetrically determined concentrations of the prepared standards. This gives the instrument analyte values to regress against corresponding intensities during the modeling stage. The regression equation correlates the concentrations of an analyte with its net intensity.

The calibration equation is developed using a least squares fit regression analysis. After the regression terms to be used in the equation are defined, a mathematical equation can be developed to calculate the analyte concentration in an unknown sample. In some FPXRF instruments, the software of the instrument calculates the regression equation. The software uses calculated intercept and slope values to form a multiterm equation. In conjunction with the software in the instrument, the operator can adjust the multiterm equation to minimize interelement interferences and optimize the intensity calibration curve.

It is possible to define up to six linear or nonlinear terms in the regression equation. Terms can be added and deleted to optimize the equation. The goal is to produce an equation with the smallest regression error and the highest correlation coefficient. These values are automatically computed by the software as the regression terms are added, deleted, or modified. It is also possible to delete data points from the regression line if these points are significant outliers or if they are heavily weighing the data. Once the regression equation has been selected for an analyte, the equation can be entered into the software for quantitation of analytes in subsequent samples. For an empirical calibration to be acceptable, the regression equation for a specific analyte should have a correlation coefficient of 0.98 or greater or meet the DQOs of the project.

In an empirical calibration, one must apply the DQOs of the project and ascertain critical or action levels for the analytes of interest. It is within these concentration ranges or around these action levels that the FPXRF instrument should be calibrated most accurately. It may not be possible to develop a good regression equation over several orders of analyte concentration.

10.4 Compton Normalization Method: The Compton normalization method is based on analysis of a single, certified standard and normalization for the Compton peak. The Compton peak is produced from incoherent backscattering of x-ray radiation from the excitation source and is present in the spectrum of every sample. The Compton peak intensity changes with differing matrices. Generally, matrices dominated by lighter elements produce a larger Compton peak, and those dominated by heavier elements produce a smaller Compton peak. Normalizing to the Compton peak can reduce problems with varying matrix effects among samples. Compton normalization is similar to the use of internal standards in organics analysis. The Compton normalization method may not be effective when analyte concentrations exceed a few percent.

The certified standard used for this type of calibration could be an NIST SRM such as 2710 or 2711. The SRM must be a matrix similar to the samples and must contain the analytes of interests at concentrations near those expected in the samples. First, a response factor has to be determined for each analyte. This factor is calculated by dividing the net peak intensity by the analyte concentration. The net peak intensity is gross intensity corrected for baseline interference. Concentrations of analytes in samples are then determined by multiplying the baseline corrected analyte signal intensity by the normalization factor and by the response factor. The normalization factor is the quotient of the baseline corrected Compton K_{α} peak intensity of the SRM divided by that of the samples. Depending on the FPXRF instrument used, these calculations may be done manually or by the instrument software.

11.0 PROCEDURE

- 11.1 Operation of the various FPXRF instruments will vary according to the manufacturers' protocols. Before operating any FPXRF instrument, one should consult the manufacturer's manual. Most manufacturers recommend that their instruments be allowed to warm up for 15 to 30 minutes before analysis of samples. This will help alleviate drift or energy calibration problems later on in analysis.
- 11.2 Each FPXRF instrument should be operated according to the manufacturer's recommendations. There are two modes in which FPXRF instruments can be operated: in situ and intrusive. The in situ mode involves analysis of an undisturbed soil sediment or sample. Intrusive analysis involves collection and preparation of a soil or sediment sample before analysis. Some FPXRF instruments can operate in both modes of analysis, while others are designed to operate in only one mode. The two modes of analysis are discussed below.
- 11.3 For in situ analysis, one requirement is that any large or nonrepresentative debris be removed from the soil surface before analysis. This debris includes rocks, pebbles, leaves, vegetation, roots, and concrete. Another requirement is that the soil surface be as smooth as possible so that the probe window will have good contact with the surface. This may require some leveling of the surface with a stainless-steel trowel. During the study conducted to provide data for this method, this modest amount of sample preparation was found to take less than 5 minutes per sample location. The last requirement is that the soil or sediment not be saturated with water. Manufacturers state that their FPXRF instruments will perform adequately for soils with moisture contents of 5 to 20 percent but will not perform well for saturated soils, especially if ponded water exists on the surface. Another recommended technique for *in situ* analysis is to tamp the soil to increase soil density and compactness for better repeatability and representativeness. This condition is especially important for heavy element analysis, such as barium. Source count times for in situ analysis usually range from 30 to 120 seconds, but source count times will vary among instruments and depending on required detection limits.

- 11.4 For intrusive analysis of surface or sediment, it is recommended that a sample be collected from a 4- by 4-inch square that is 1 inch deep. This will produce a soil sample of approximately 375 grams or 250 cm³, which is enough soil to fill an 8-ounce jar. The sample should be homogenized, dried, and ground before analysis. The sample can be homogenized before or after drying. The homogenization technique to be used after drying is discussed in Section 4.2. If the sample is homogenized before drying, it should be thoroughly mixed in a beaker or similar container, or if the sample is moist and has a high clay content, it can be kneaded in a plastic bag. One way to monitor homogenization when the sample is kneaded in a plastic bag is to add sodium, fluorescein dye to the sample. After the moist sample has been homogenized, it is examined under Dye an ultraviolet light to assess the distribution of sodium fluorescein throughout the sample. If the fluorescent dye is evenly distributed in the sample, homogenization is considered complete; if the dye is not evenly distributed, mixing should continue until the sample has been thoroughly homogenized. During the study conducted to provide data for this method, the homogenization procedure using the fluorescein dye required 3 to 5 minutes per sample. As demonstrated in Sections 13.5 and 13.7, homogenization has the greatest impact on the reduction of sampling variability. It produces little or no contamination. Often, it can be used without the more labor intensive steps of drying, grinding, and sieving given in Sections 11.5 and 11.6. Of course, to achieve the best data quality possible all four steps must be followed.
- 11.5 Once the soil or sediment sample has been homogenized, it should be dried. This can be accomplished with a toaster oven or convection oven. A small aliquot of the sample (20 to 50 grams) is placed in a suitable container for drying. The sample should be dried for 2 to 4 hours in the convection or toaster oven at a temperature not greater than 150°C. Microwave drying is not a recommended procedure. Field studies have shown that microwave drying can increase variability between the FPXRF data and confirmatory analysis. High levels of metals in a sample can cause arcing in the microwave oven, and sometimes slag forms in the sample. Microwave oven drying can also melt plastic containers used to hold the sample.
- The homogenized dried sample material should be ground with a mortar and pestle and passed through a 60-mesh sieve to achieve a uniform particle size. Sample grinding should continue until at least 90 percent of the original sample passes through the sieve. The grinding step normally takes an average of 10 minutes per sample. An aliquot of the sieved sample should then be placed in a 31 0-mm polyethylene sample cup (or equivalent) for analysis. The sample cup should be one-half to three-quarters full at a minimum. The sample cup should be covered with a 2.5 µm Mylar (or equivalent) film for analysis. The rest of the soil sample should be placed in a jar, labeled, and archived for possible confirmation analysis. All equipment including the mortar, pestle, and sieves must be thoroughly cleaned so that any cross-contamination is below the MDLs of the procedure or DQOs of the analysis.

12.0 DATA ANALYSIS AND CALCULATIONS

Most FPXRF instruments have software capable of storing all analytical results and spectra. The results are displayed in parts per million and can be downloaded to a PC, which can provide a hard copy printout. Individual measurements that are smaller than three times their associated SD should not be used for quantitation.

13.0 METHOD PERFORMANCE

This section discusses four performance factors, field-based method detection limits, precision, accuracy, and comparability to EPA-approved methods. The numbers presented in Tables 4 through 9 were generated from data obtained from six FPXRF instruments. The soil samples analyzed by the six FPXRF instruments were collected from two sites in the United States.

PLEA

6.3.3

Without

6.3.3

Prep. 6.3.4 The soil samples contained several of the target analytes at concentrations ranging from nondetect to tens of thousands of mg/kg.

- 13.2 The six FPXRF instruments included the TN 9000 and TN Lead Analyzer manufactured by TN Spectrace; the X-MET 920 with a SiLi detector and X-MET 920 with a gas-filled proportional detector manufactured by Metorex, Inc.; the XL Spectrum Analyzer manufactured by Niton; and the MAP Spectrum Analyzer manufactured by Scitec. The TN 9000 and TN Lead Analyzer both have a Hgl₂ detector. The TN 9000 utilized an Fe-55, Cd-109, and Am-241 source. The TN Lead Analyzer had only a Cd-109 source. The X-Met 920 with the SiLi detector had a Cd-109 and Am-241 source. The X-MET 920 with the gas-filled proportional detector had only a Cd-109 source. The XL Spectrum Analyzer utilized a silicon pin-diode detector and a Cd-109 source. The MAP Spectrum Analyzer utilized a solid-state silicon detector and a Cd-109 source.
- 13.3 All data presented in Tables 4 through 9 were generated using the following calibrations and source count times. The TN 9000 and TN Lead Analyzer were calibrated using fundamental parameters using NIST SRM 2710 as a calibration check sample. The TN 9000 was operated using 100, 60, and 60 second count times for the Cd-109, Fe-55, and Am-241 sources, respectively. The TN Lead analyzer was operated using a 60 second count time for the Cd-109 source. The X-MET 920 with the Si(Li) detector was calibrated using fundamental parameters and one well characterized site-specific soil standard as a calibration check. It used 140 and 100 second count times for the Cd-109 and Am-241 sources, respectively. The X-MET 920 with the gas-filled proportional detector was calibrated empirically using between 10 and 20 well characterized site-specific soil standards. It used 120 second times for the Cd-109 source. The XL Spectrum Analyzer utilized NIST SRM 2710 for calibration and the Compton peak normalization procedure for quantitation based on 60 second count times for the Cd-109 source. The MAP Spectrum Analyzer was internally calibrated by the manufacturer. The calibration was checked using a well-characterized site-specific soil standard. It used 240 second times for the Cd-109 source.
- 13.4 Field-Based Method Detection Limits: The field-based method detection limits are presented in Table 4. The field-based method detection limits were determined by collecting ten replicate measurements on site-specific soil samples with metals concentrations 2 to 5 times the expected method detection limits. Based on these ten replicate measurements, a standard deviation on the replicate analysis was calculated. The method detection limits presented in Table 4 are defined as 3 times the standard deviation for each analyte.

The field-based method detection limits were generated by using the count times discussed earlier in this section. All the field-based method detection limits were calculated for soil samples that had been dried and ground and placed in a sample cup with the exception of the MAP Spectrum Analyzer. This instrument can only be operated in the in situ mode, meaning the samples were moist and not ground.

Some of the analytes such as cadmium, mercury, silver, selenium, and thorium were not detected or only detected at very low concentrations such that a field-based method detection limit could not be determined. These analytes are not presented in Table 4. Other analytes such as calcium, iron, potassium, and titanium were only found at high concentrations (thousands of mg/kg) so that reasonable method detection limits could not be calculated. These analytes also are not presented in Table 4.

13.5 Precision Measurements: The precision data is presented in Table 5. Each of the six FPXRF instruments performed 10 replicate measurements on 12 soil samples that had analyte concentrations ranging from nondetects to thousands of mg/kg. Each of the 12 soil samples underwent 4 different preparation techniques from in situ (no preparation) to dried and ground in a

sample cup. Therefore, there were 48 precision data points for five of the instruments and 24 precision points for the MAP Spectrum Analyzer. The replicate measurements were taken using the source count times discussed at the beginning of this section.

For each detectable analyte in each precision sample a mean concentration, standard deviation, and RSD was calculated for each analyte. The data presented in Table 5 is an average RSD for the precision samples that had analyte concentrations at 5 to 10 times the MDL for that analyte for each instrument. Some analytes such as mercury, selenium, silver, and thorium were not detected in any of the precision samples so these analytes are not listed in Table 5. Some analytes such as cadmium, nickel, and tin were only detected at concentrations near the MDLs so that an RSD value calculated at 5 to 10 times the MDL was not possible.

One FPXRF instrument collected replicate measurements on an additional nine soil samples to provide a better assessment of the effect of sample preparation on precision. Table 6 shows these results. The additional nine soil samples were comprised of three from each texture and had analyte concentrations ranging from near the detection limit of the FPXRF analyzer to thousands of mg/kg. The FPXRF analyzer only collected replicate measurements from three of the preparation methods; no measurements were collected from the *in situ* homogenized samples. The FPXRF analyzer conducted five replicate measurements of the in situ field samples by taking measurements at five different points within the 4-inch by 4-inch sample square. Ten replicate measurements were collected for both the intrusive undried and unground and intrusive dried and ground samples contained in cups. The cups were shaken between each replicate measurement.

Table 6 shows that the precision dramatically improved from the in situ to the intrusive measurements. In general there was a slight improvement in precision when the sample was dried and ground. Two factors caused the precision for the in situ measurements to be poorer. The major factor is soil heterogeneity. By moving the probe within the 4-inch by 4-inch square, measurements of different soil samples were actually taking place within the square. Table 6 illustrates the dominant effect of soil heterogeneity. It overwhelmed instrument precision when the FPXRF analyzer was used in this mode. The second factor that caused the RSD values to be higher for the in situ measurements is the fact that only five versus ten replicates were taken. A lesser number of measurements caused the standard deviation to be larger which in turn elevated the RSD values.

13.6 Accuracy Measurements: Five of the FPXRF instruments (not including the MAP Spectrum Analyzer) analyzed 18 SRMs using the source count times and calibration methods given at the beginning of this section. The 18 SRMs included 9 soil SRMs, 4 stream or river sediment SRMs, 2 sludge SRMs, and 3 ash SRMs. Each of the SRMs contained known concentrations of certain target analytes. A percent recovery was calculated for each analyte in each SRM for each FPXRF instrument. Table 7 presents a summary of this data. With the exception of cadmium, chromium, and nickel, the values presented in Table 7 were generated from the 13 soil and sediment SRMs only. The 2 sludge and 3 ash SRMs were included for cadmium, chromium, and nickel because of the low or nondetectable concentrations of these three analytes in the soil and sediment SRMs.

Only 12 analytes are presented in Table 7. These are the analytes that are of environmental concern and provided a significant number of detections in the SRMs for an accuracy assessment. No data is presented for the X-MET 920 with the gas-filled proportional detector. This FPXRF instrument was calibrated empirically using site-specific soil samples. The percent recovery values from this instrument were very sporadic and the data did not lend itself to presentation in Table 7.

Table 8 provides a more detailed summary of accuracy data for one FPXRF instrument (TN 9000) for the 9 soil SRMs and 4 sediment SRMs. Table 8 shows the certified value, measured

value, and percent recovery for five analytes. These analytes were chosen because they are of environmental concern and were most prevalently certified for in the SRM and detected by the FPXRF instrument. The first nine SRMs are soil and the last 4 SRMs are sediment. Percent recoveries for the four NIST SRMs were often between 90 and 110 percent for all analytes.

13.7 Comparability: Comparability refers to the confidence with which one data set can be compared to another. In this case, FPXRF data generated from a large study of six FPXRF instruments was compared to SW-846 Methods 3050 and 6010 which are the standard soil extraction for metals and analysis by inductively coupled plasma. An evaluation of comparability was conducted by using linear regression analysis. Three factors were determined using the linear regression. These factors were the y-intercept, the slope of the line, and the coefficient of determination (r^2) .

As part of the comparability assessment, the effects of soil type and preparation methods were studied. Three soil types (textures) and four preparation methods were examined during the study. The preparation methods evaluated the cumulative effect of particle size, moisture, and homogenization on comparability. Due to the large volume of data produced during this study, linear regression data for six analytes from only one FPXRF instrument is presented in Table 9. Similar trends in the data were seen for all instruments.

Table 9 shows the regression parameters for the whole data set, broken out by soil type, and by preparation method. The soil types are as follows: soil 1—sand; soil 2—loam; and soil 3—silty clay. The preparation methods are as follows: preparation 1—in situ in the field; preparation 2—in situ, sample collected and homogenized; preparation 3—intrusive, with sample in a sample cup but sample still wet and not ground; and preparation 4—sample dried, ground, passed through a 40-mesh sieve, and placed in sample cup.

For arsenic, copper, lead, and zinc, the comparability to the confirmatory laboratory was excellent with r^2 values ranging from 0.80 to 0.99 for all six FPXRF instruments. The slopes of the regression lines for arsenic, copper, lead, and zinc, were generally between 0.90 and 1.00 indicating the data would need to be corrected very little or not at all to match the confirmatory laboratory data. The r^2 values and slopes of the regression lines for barium and chromium were not as good as for the other for analytes, indicating the data would have to be corrected to match the confirmatory laboratory.

Table 9 demonstrates that there was little effect of soil type on the regression parameters for any of the six analytes. The only exceptions were for barium in soil 1 and copper in soil 3. In both of these cases, however, it is actually a concentration effect and not a soil effect causing the poorer comparability. All barium and copper concentrations in soil 1 and 3, respectively, were less than 350 mg/kg.

Table 9 shows there was a preparation effect on the regression parameters for all six analytes. With the exception of chromium, the regression parameters were primarily improved going from preparation 1 to preparation 2. In this step, the sample was removed from the soil surface, all large debris was removed, and the sample was thoroughly homogenized. The additional two preparation methods did little to improve the regression parameters. This data indicates that homogenization is the most critical factor when comparing the results. It is essential that the sample sent to the confirmatory laboratory match the FPXRF sample as closely as possible.

Section 11.0 of this method discusses the time necessary for each of the sample preparation techniques. Based on the data quality objectives for the project, an analyst must decide if it is worth the extra time required to dry and grind the sample for small improvements in comparability.

Homogenization requires 3 to 5 minutes. Drying the sample requires one to two hours. Grinding and sieving requires another 10 to 15 minutes per sample. Lastly, when grinding and sieving is conducted, time must be allotted to decontaminate the mortars, pestles, and sieves. Drying and grinding the samples and decontamination procedures will often dictate that an extra person be on site so that the analyst can keep up with the sample collection crew. The cost of requiring an extra person on site to prepare samples must be balanced with the gain in data quality and sample throughput.

- 13.8 The following documents may provide additional guidance and insight on this method and technique:
 - 13.8.1 Hewitt, A.D. 1994. "Screening for Metals by X-ray Fluorescence Spectrometry/Response Factor/Compton K_{α} Peak Normalization Analysis." *American Environmental Laboratory*. Pages 24-32.
 - 13.8.2 Piorek, S., and J.R. Pasmore. 1993. "Standardless, In Situ Analysis of Metallic Contaminants in the Natural Environment With a PC-Based, High Resolution Portable X-Ray Analyzer." *Third International Symposium on Field Screening Methods for Hazardous Waste and Toxic Chemicals.* Las Vegas, Nevada. February 24-26, 1993. Volume 2, Pages 1135-1151.

14.0 POLLUTION PREVENTION

- 14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.
- 14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202) 872-4477.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

1. Metorex. X-MET 920 User's Manual.

- 2. Spectrace Instruments. 1994. Energy Dispersive X-ray Fluorescence Spectrometry: An Introduction.
- 3. TN Spectrace. Spectrace 9000 Field Portable/Benchtop XRF Training and Applications Manual.
- 4. Unpublished SITE data, recieved from PRC Environment Management, Inc.
- 17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The pages to follow contain Tables 1 through 9 and a method procedure flow diagram.

TABLE 1
INTERFERENCE FREE DETECTION LIMITS

Analyte	Chemical Abstract Series Number	Detection Limit in Quartz Sand (milligrams per kilogram)
Antimony (Sb)	7440-36-0	40
Arsenic (As)	7440-38-0	40
Barium (Ba)	7440-39-3	20
Cadmium (Cd)	7440-43-9	100
Calcium (Ca)	7440-70-2	70
Chromium (Cr)	7440-47-3	150
Cobalt (Co)	7440-48-4	60
Copper (Cu)	7440-50-8	50
Iron (Fe)	7439-89-6	60
Lead (Pb)	7439-92-1	20
Manganese (Mn)	7439-96-5	70
Mercury (Hg)	7439-97-6	30
Molybdenum (Mo)	7439-93-7	10
Nickel (Ni)	7440-02-0	50
Potassium (K)	7440-09-7	200
Rubidium (Rb)	7440-17-7	10
Selenium (Se)	7782-49-2	40
Silver (Ag)	7440-22-4	70
Strontium (Sr)	7440-24-6	10
Thallium (TI)	7440-28-0	20
Thorium (Th)	7440-29-1	10
Tin (Sn)	7440-31-5	60
Titanium (Ti)	7440-32-6	50
Vanadium (V)	7440-62-2	50
Zinc (Zn)	7440-66-6	50
Zirconium (Zr)	7440-67-7	10

Source: References 1, 2, and 3

TABLE 2
SUMMARY OF RADIOISOTOPE SOURCE CHARACTERISTICS

Source	Activity (mCi)	Half-Life (Years)	Excitation Energy (keV)	Elemental Analysis Range	
Fe-55	20 -50	2.7	5.9	Sulfur to Chromium K I Molybdenum to Barium L I	
Cd-109	5-30	1.3	22.1 and 87.9	Calcium to Rhodium K L Tantalum to Lead K L Barium to Uranium L L	
Am-241	5-30	458	26.4 and 59.6	Copper to Thulium Tungsten to Uranium	K Lines L Lines
Cm-244	60-100	17.8	14.2	Titanium to Selenium Lanthanum to Lead	K Lines L Lines

Source: Reference 1, 2, and 3

TABLE 3
SUMMARY OF X-RAY TUBE SOURCE CHARACTERISTICS

Anode Material	Recommended Voltage Range (kV)	K-alpha Emission (keV)	Elemental Analysis Range		
Cu	18-22	8.04	Potassium to Cobalt Silver to Gadolinium	K Lines L Lines	
Мо	40-50	17.4	Cobalt to Yttrium Europium to Radon	K Lines L Lines	
Ag	50-65	22.1	Zinc to Technicium Ytterbium to Neptunium	K Lines L Lines	

Source: Reference 4

Notes: The sample elements excited are chosen by taking as the lower limit the same ratio of excitation line energy to element absorption edge as in Table 2 (approximately 0.45) and the requirement that the excitation line energy be above the element absorption edge as the upper limit (L2 edges used for L lines). K-beta excitation lines were ignored.

TABLE 4
FIELD-BASED METHOD DETECTION LIMITS (mg/kg)^a

			lı	nstrument		
Analyte	TN 9000	TN Lead Analyzer	X-MET 920 (SiLi Detector)	X-MET 920 (Gas-Filled Detector)	XL Spectrum Analyzer	MAP Spectrum Analyzer
Antimony	55	NR	NR	NR	NR	. NR
Arsenic	60	50	55	50	110	225
Barium	60	NR	30	400	NR	NR
Chromium	200	460	210	110	900	NR
Cobalt	330	NR	NR	NR	NR	NR
Copper	85	115	75	100	125	525
Lead	45	40	45	100	75	165
Manganese	240	340	NR	NR	NR	NR
Molybdenum	25	NR	NR	NR	30	NR
Nickel	100	NR	NA	NA	NA	NR
Rubidium	30	NR	NR	NR	45	NR
Strontium	35	NR	NR	NR	40	NR
Tin	85	NR	NR	NR	NR	NR
Zinc	80	95	70	NA	110	NA
Zirconium	40	NR	NR	. NR	25	NR

^a MDLs are related to the total number of counts taken. See Section 13.3 for count times used to generate this table.

NR Not reported.

NA Not applicable; analyte was reported but was not at high enough concentrations for method detection limit to be determined.

TABLE 5
PRECISION

Analyte		Average R	elative Standar at 5 to 10	d Deviation for Times the MDL		ent
	TN 9000	TN Lead Analyzer	X-MET 920 (SiLi Detector)	X-MET 920 (Gas-Filled Detector)	XL Spectrum Analyzer	MAP Spectrum Analyzer
Antimony	6.54	NR	NR	NR	NR	NR
Arsenic	5.33	4.11	3.23	1.91	12.47	6.68
Barium	4.02	NR	3.31	5.91	NR	NR
Cadmium	29.84ª	NR	24.80ª	NR	NR	NR
Calcium	2.16	NR	NR	NR	NR	NR
Chromium	22.25	25.78	22.72	3.91	30.25	NR
Cobalt	33.90	NR	NR	NR	NR	NR
Copper	7.03	9.11	8.49	9.12	12.77	14.86
Iron	1.78	1.67	1.55	NR	2.30	NR
Lead	6.45	5.93	5.05	7.56	6.97	12.16
Manganese	27.04	24.75	NR	NR	NR	NR
Molybdenum	6.95	NR	NR	NR	12.60	NR
Nickel	30.85ª	NR	24.92	20.92°	NA	NR
Potassium	3.90	NR	NR	NR	NR	NR
Rubidium	13.06	NR	NR	NR	32.69ª	NR
Strontium	4.28	NR	NR	NR	8.86	NR
Tin	24.32ª	NR	NR	NR	NR	NR
Titanium	4.87	NR	NR	NR	NR	NR
Zinc	7.27	7.48	4.26	2.28	10.95	0.83
Zirconium	3.58	NR	NR	NR	6.49	NR

These values are biased high because the concentration of these analytes in the soil samples was near the detection limit for that particular FPXRF instrument.

NR Not reported.

NA Not applicable; analyte was reported but was below the method detection limit.

TABLE 6
PRECISION AS AFFECTED BY SAMPLE PREPARATION

	Average Relative S	Standard Deviation for Each	Preparation Method
Analyte	In Situ-Field	Intrusive- Undried and Unground	Intrusive- Dried and Ground
Antimony	30.1	15.0	14.4
Arsenic	22.5	5.36	3.76
Barium	17.3	3.38	2.90
Cadmiuma	41.2	30.8	28.3
Calcium	17.5	1.68	1.24
Chromium	17.6	28.5	21.9
Cobalt	28.4	31.1	28.4
Copper	26.4	10.2	7.90
Iron	10.3	1.67	1.57
Lead	25.1	8.55	6.03
Manganese	40.5	12.3	13.0
Mercury	ND	ND	ND
Molybdenum	21.6	20.1	19.2
Nickel	29.8	20.4	18.2
Potassium	18.6	3.04	2.57
Rubidium	29.8	16.2	18.9
Selenium	ND	20.2	19.5
Silver*	31.9	31.0	29.2
Strontium	15.2	3.38	3.98
Thallium	39.0	16.0	19.5
Thorium	NR	NR	NR
Tin	ND	14.1	15.3
Titanium	13.3	4.15	3.74
Vanadium	NR	NR	NR
Zinc	26.6	13.3	11.1
Zirconium	20.2	5.63	5.18

ND Not detected.

NR Not reported.

These values may be biased high because the concentration of these analytes in the soil samples was near the detection limit.

TABLE 7
ACCURACY

							lı	nstrume	nt								
		TN 90	000			TN Lead	Analyzer		X-N	MET 920 (S	SiLi Detec	ctor)		XL Spectrum Analyzer			
Analyte	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec	SD	n	Range of % Rec.	Mean % Rec.	SD	
Sb	2	100-149	124.3	NA			***	**		70							
As	5	68-115	92.8	17.3	5	44-105	83.4	23.2	4	9.7-91	47.7	39.7	5	38-535	189.8	206	
Ва	9	98-198	135.3	36.9					9	18-848	168.2	262					
Cd	2	99-129	114.3	NA					6	81-202	110.5	45.7			**		
Сг	2	99-178	138.4	NA		***			7	22-273	143.1	93.8	3	98-625	279.2	300	
Cu	8	61-140	95.0	28.8	6	38-107	79.1	27.0	11	10-210	111.8	72.1	8	95-480	203.0	147	
Fe	6	78-1 <u>5</u> 5	103.7	26.1	6	89-159	102.3	28.6	6	48-94	80.4	16.2	· 6	26-187	108.6	52.9	
Pb	11	66-138	98.9	19.2	11	68-131	97.4	18.4	12	23-94	72.7	20.9	13	80-234	107.3	39.9	
Mn	4	81-104	93.1	9.70	3	92-152	113.1	33.8									
Ni	3	99-122	109.8	12.0		6 pa							3	57-123	87.5	33.5	
Sr	8	110-178	132.6	23.8		•••							7	86-209	125.1	39.5	
Zn	11	41-130	94.3	24.0	10	81-133	100.0	19.7	12	46-181	106.6	34.7	11	31-199	94.6	42.5	

n Number of samples that contained a certified value for the analyte and produced a detectable concentration from the FPXRF instrument.

SD Standard deviation.

NA Not applicable; only two data points, therefore, a SD was not calculated.

%Rec. Percent recovery.

-- No data.

TABLE 8
ACCURACY FOR TN 9000*

Standard		Arsenic			Barium			Copper			Lead	.		Zinc	
Reference Material	Cert. Conc.	Meas. Conc.	%Rec.												
RTC CRM-021	24.8	ND	NA	586	1135	193.5	4792	2908	60.7	144742	149947	103.6	546	224	40.9
RTC CRM-020	397	429	92.5	22.3	ND	NA	753	583	77.4	5195	3444	66.3	3022	3916	129.6
BCR CRM 143R							131	105	80.5	180	206	114.8	1055	1043	99.0
BCR CRM 141							32.6	ND	NA	29.4	ND	NA	81.3	ND	NA
USGS GXR-2	25.0	ND	NA	2240	2946	131.5	76.0	106	140.2	690	742	107.6	530	596	112.4
USGS GXR-6	330	294	88.9	1300	2581	198.5	66.0	ND	. NA	101	80.9	80.1	118	ND	NA
NIST 2711	105	104	99.3	726	801	110.3	114	ND	NA	1162	1172	100.9	350	333	94.9
NIST 2710	626	722	115.4	707	782	110.6	2950	2834	96.1	5532	5420	98.0	6952	6476	93.2
NIST 2709	17.7	ND	NA	968	950	98.1	34.6	ND	NA	18.9	ND	NA	106	98.5	93.0
NIST 2704	23.4	ND	NA	414	443	107.0	98.6	105	106.2	161	167	103.5	438	427	97.4
CNRC PACS-1	211	143	67.7		772	NA	452	302	、66.9	404	332	82.3	824	611	74.2
SARM-51				335	466	139.1	268	373	139.2	5200	7199	138.4	2200	2676	121.6
SARM-52				410	527	128.5	219	193	88.1	1200	1107	92.2	264	215	81.4

^a All concentrations in milligrams per kilogram.

%Rec. Percent recovery.
ND Not detected.

NA Not applicable.

-- No data.

TABLE 9 REGRESSION PARAMETERS FOR COMPARABILITY¹

		Arse	enic			Barlum				Copper			
	n	r²	Int.	Slope	n	r ²	Int.	Slope	n	r²	Int.	Slope	
All Data	824	0.94	1.62	0.94	1255	0.71	60.3	0.54	984	0.93	2.19	0.93	
Soil 1	368	0.96	1.41	0.95	393	0.05	42.6	0.11	385	0.94	1.26	0.99	
Soil 2	453	0.94	1.51	0.96	462	0.56	30.2	0.66	463	0.92	2.09	0.95	
Soil 3					400	0.85	44.7	0.59	136	0.46	16.60	0.57	
Prep 1	207	0.87	2.69	0.85	312	0.64	53.7	0.55	256	0.87	3.89	0.87	
Prep 2	208	0.97	1.38	0.95	315	0.67	64.6	0.52	246	0.96	2.04	0.93	
Prep 3	204	0.96	1.20	0.99	215	0.78	64.6	0.53	236	0.97	1.45	0.99	
Prep 4	205	C.96	1.45	0.98	313	0.81	58.9	0.55	246	0.96	1.99	0.96	
	T	Lead			Zinc								
II.		LE	au		1		nc		<u> </u>	Chro	mium		
	n	r ²	Int.	Slope	n		nc Int.	Slope	n	r ²	Int.	Slope	
All Data	n 1205			Slope 0.95	n 1103		 	Slope 0.95	n 280	7	T	Slope 0.42	
All Data Soil 1		r²	Int.	 	 	r²	Int.	 	 	r ²	Int.		
<u> </u>	1205	r ²	Int. 1.66	0.95	1103	r² 0.89	Int. 1.86	0.95	280	r² 0.70	Int. 64.6	0.42	
Soil 1	1205 357	0.92 0.94	Int. 1.66 1.41	0.95 0.96	1103 329	r ² 0.89 0.93	Int. 1.86 1.78	0.95 0.93	280	r² 0.70	Int. 64.6	0.42	
Soil 1 Soil 2	1205 357 451	r ² 0.92 0.94 0.93	Int. 1.66 1.41 1.62	0.95 0.96 0.97	1103 329 423	r ² 0.89 0.93 0.85	Int. 1.86 1.78 2.57	0.95 0.93 0.90	280	0.70 —	Int. 64.6 —	0.42 — —	
Soil 1 Soil 2 Soil 3	1205 357 451 397	r ² 0.92 0.94 0.93 0.90	1.66 1.41 1.62 2.40	0.95 0.96 0.97 0.90	1103 329 423 351	r ² 0.89 0.93 0.85 0.90	Int. 1.86 1.78 2.57 1.70	0.95 0.93 0.90 0.98	280 — — — 186	72 0.70 0.66	Int. 64.6 —————————————————————————————————	0.42 — — 0.50	
Soil 1 Soil 2 Soil 3 Prep 1	1205 357 451 397 305	r ² 0.92 0.94 0.93 0.90 0.80	Int. 1.66 1.41 1.62 2.40 2.88	0.95 0.96 0.97 0.90 0.86	1103 329 423 351 286	r ² 0.89 0.93 0.85 0.90 0.79	Int. 1.86 1.78 2.57 1.70 3.16	0.95 0.93 0.90 0.98 0.87	280 ————————————————————————————————————	r ² 0.70 0.66 0.80	Int. 64.6 —————————————————————————————————	0.42 — — 0.50 0.43	

Source: Reference 4

Log-transformed data Number of data points n

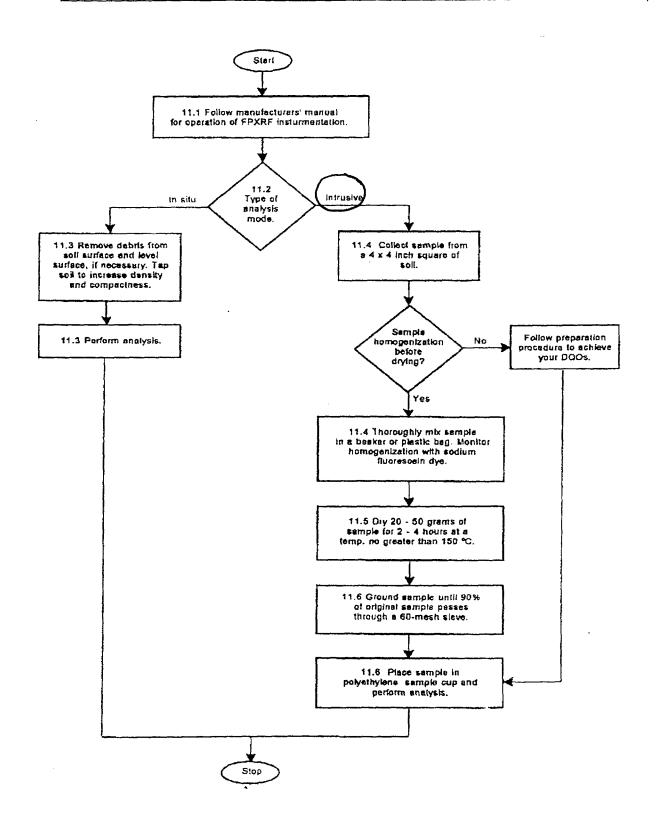
Coefficient of determination

Y-intercept Int.

No applicable data

METHOD 6200

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT



Page 1 of 6

LOW CONCENTRATION SOIL CONTAMINANT CHARACTERIZATION USING EDXRF ANALYSIS

AR. Harding

INTRODUCTION

Effective assessment and remediation of hazardous waste sites dictates that analytical methodologies be developed which assist in the evaluation of site contamination and simultaneously make efficient use of sampling time and resources (1). Optimally, a technique would provide on-site personnel with immediate and accurate information concerning the identity and concentration of inorganic soil contaminants (2).

Inorganic pollutants can be readily determined in contaminated soils with energy dispersive X-ray fluorescence spectrometry (EDXRF) using a thermoelectrically cooled Si(Li) detector (3). A field mobile laboratory van or trailer can accommodate the EDXRF system because the electrically cooled detector, which provides high resolution EDXRF spectra, does not require cryogenic cooling. Soil sample preparation for EDXRF analysis is minimal, therefore, short turnaround times are realized between sampling and reporting results.

This report will describe an EDXRF method developed to determine four inorganic soil contaminants: lead, arsenic, zinc, and cadmium at four sampling depths. The EDXRF results for approximately one hundred eighty soil samples will be compared to results obtained for sample splits submitted for analysis at an independent laboratory. Evaluation of low concentration arsenic detectability with elevated lead concentrations in these samples will be discussed. Accuracy and precision of the EDXRF method will also be compared to the independent methods using a standard reference material and soil samples submitted in triplicate to both laboratories.

EXPERIMENTAL

The field mobile EDXRF spectrometer used in this work was a Spectrace 6000 (Spectrace Instruments, Inc., Mountain View, CA). The EDXRF system consists of three modules: the spectrometer, the control/pulse processing electronics, and the data analysis computer. The compact size and weight (90 lbs.) of the modules permits installation of the system in a laboratory trailer or van.

The bench top spectrometer module, which can accommodate a single soil sample, is powered by 110 V line or generator feed. The excitation source used is a low powered Rh anode X-ray tube (50 KV, 0.35 mA (17 W) maximum output) positioned at a 45° incident angle to the sample. Three primary radiation filters permit optimum spectral acquisition conditions to be computer selected.

The thermoelectrically cooled Si(Li) X-ray detector is mounted at a 45° take-off angle in an inverted geometry with respect to the sample. The 20 mm2 Si(Li) crystal, which is protected by a 0.5 mil Be window, is cooled to -90°C for operation using a multi-stage thermoelectric (Peltier effect) cooler. The 300 watts produced at the detector heat sink are dissipated by forced ambient air. Thermoelectrically cooled detectors provide typical resolutions of 185 eV (Mn Ka).

A card cage module is interfaced between the spectrometer and a personal computer. The card cage components include the detector high voltage supply, the pulse processing electronics, and the control circuit board for the EDXRF spectrometer. The data analysis software executed on the PC is capable of either a fundamental parameters or empirical data treatment scheme using a combination of standard reference materials and/or site specific standards.

Page 3 of 6

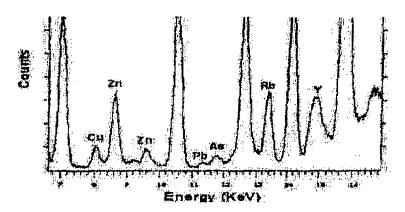


Figure 1. Mid Z spectrum of a soil sample containing 1100 ppm Pb, 729 ppm Zn, and 125 ppm As. Full scale on the y-axis is 2,000 counts.

The soil characterization method was standardized using four standard reference materials (SRM): NBS 1648 (urban particulate); NBS 2704 (river sediment); SO-1 and SO-3, two soil standards available from the Canada Centre for Mineral and Energy Technology. Standards labeled NBS are available from the National Institute for Standards and Technology (NIST). These SRMs have certified concentrations of Fe, Mn, Cu, Zn, Pb, and Cd.

A fundamental parameters (FP) method (5) was employed as the data treatment scheme and used certified concentrations of Fe, Mn, Cu, Zn, Pb, and Cd in the four standard materials. To compute instrumental sensitivity (emission peak counts per second per ppm), the balance of the standard was assumed to be comprised of SiO2 to account for the contribution of the matrix on the measured analyte X-ray intensity. The balance component SiO5 was selected to mimic the concentration of Si and 0 in typical soils, approximately 24% Si and 45% 0. Since none of the selected SRMs contain arsenic, As sensitivity (cps/ppm) was determined using a fundamental parameters theoretical calculation based on the computed Zn sensitivity. Table 2 lists the analyte sensitivities computed by the FP method.

ANALYTE	SENSIT(VITY (cps/ppm)	LLD (ppm)
Mn	0.010	21
Fe	0.015	19
Cu	0.046	26
Zn	0.067	19
Pb	0.084	7
As	0.132	12
Cd	0.107	4

Table 2. Sensitivity and lower limits of detection for the analytes of interest.

There are some advantages to using an FP method for standardization compared to site specific soil standards. The FP method can use readily available, well-characterized SRMs to measure analyte sensitivities. Site specific soil standards, by contrast, are usually collected with a separate sampling mobilization. The FP method standardized with SRMs can provide accurate analyte concentrations to be determined in samples with fairly wide matrix variations without restandardization, unlike methods incorporating site specific standards.

Page 4 of 6

RESULTS

Table 2 lists the lower limits of detection determined using the two sets of spectral acquisition conditions (Table 1). The pertinent equation is: LLD (ppm) = 3*sqrt(Ib)/m*sqrt(T), where Ib is the background intensity (cps), m is the analyte sensitivity (cps/ppm), and T is the acquisition livetime in seconds (6). Calculated LLD values are dependent upon spectrum acquisition times, sample matrix, and excitation conditions. The conditions in Table 1 were selected to optimize the Pb and Cd spectral regions. Improved LLDs are possible with EDXRF using longer spectrum acquisition livetimes and optimized excitation conditions for selected spectral regions.

Results for the determination of four analytes by EDXRF in 180 samples (43 cores at 4 levels, two SRMs, three samples in triplicate) were compared to independent analysis results in order to evaluate the level of agreement between the two methods. Table 3 lists the correlation plot data for the analytes in terms of actual slope, intercept, errors, and the correlation coefficient of the fit. Each analyte correlation plot included approximately 150 data points.

ANALYTE	SLOPE	INTERCEPT	CORRELATION COEFFICIENT
Pb	1.01 ± 0.03	10.0 ± 13.8	0.96
As	1.08 ± 0.05	0.98 ± 3.54	0.92
Cd	1.02 ± 0.03	3.09 ± 2.19	0.94
"Zn	1.02 ± 0.02	63.0 ± 13.6	0.98

Table 3. Correlation plot data for the four analytes of environmental interest.

As shown in Table 3, slopes of the plots for Pb, Cd, Zn, and As are within 8% of 1.00 and all correlation coefficients are greater than 0.92. The calculated slope near 1.00 and correlation coefficients greater than 0.90 indicates agreement between the two analytical techniques. Figure 2 is a plot of 94 data points in the range of 0 to 300 ppm Pb. Figure 3 is a plot of 110 EDXRF and ICP analyzed samples in the range of 0 to 100 ppm Cd and also indicates agreement between the results of the two methods.

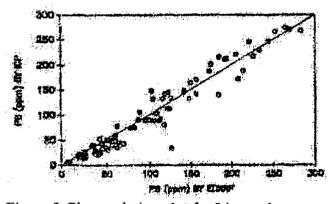


Figure 2. Pb correlation plot for 94 samples.



Page 5 of 6

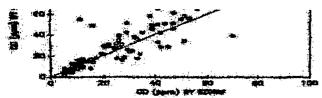


Figure 3. Cd correlation plot for 110 samples.

ACCURACY AND PRECISION

To evaluate the accuracy provided by the EDXRF method two SRMs were submitted as unknowns for EDXRF analysis as well as being submitted to the independent lab for analysis. Table 4 lists the results for SRM SO-2. EDXRF analysis of SO-2 provides results that are in good agreement with certified values. The independent ICP analysis of zinc in SO-2, however, is biased low by a factor of one-half.

Sample	Analyte	ICP	EDXRF	Certified
SO-2	Pb	19	17	21
	Zn	55	123	124

Table 4. Results of the analysis of SRM SO-2 by ICP and EDXRF methods. All values in ppm.

Precision was evaluated by submitting three samples a total of three times for independent and EDXRF analysis. Table 5 shows the results for the two methods along with the calculated standard deviation (in ppm) of the three replicate analyses. Note that Cd in sample C was only reported by EDXRF to the nearest 1 ppm and three values of 9 ppm Cd were determined, hence the zero standard deviation for the three replicates. EDXRF precision is better than 10% relative standard deviation in all but one case (As in sample C) and compares well with that provided by the independent lab.

Sample	Element	Ind. Lab	EDXRF
А	As	45 ± 4	41 ± 3
	Cd	20 ± 2	31 ± 3
	Pb	286 ± 28	312 ± 12
	Zn	185 ± 15	134 ± 10
В	As	17 ± 3	14 ± 1
	Cd	80 ± 6	58 ± 4
	Pb	141 ± 15	158 ± 3
	Zn	556 ± 39	529 ± 46
С	A s	17 ± 1	19 ± 4
	Cd	10.0 ± 0.9	9 ± 0
	Pb	117 ± 8	142 ± 14
	Zn	173 ± 26	128 ± 3

Table 6. EDXRF and independent lab results for three soil samples each analyzed in triplicate. All values in ppm

CONCLUSION

Field mobile EDXRF analysis of soils suspected of being contaminated provides information concerning the nature, extent, and magnitude of the contamination. Due to the minimal sample preparation necessary for EDXRF analysis, sampling to result turnaround time is relatively short so

Page 6 of 6

THE DESTREE

preparation necessary for EDXRF analysis, sampling to result turnaround time is relatively short so the most effective use of sampling resources is realized. EDXRF detection limits below 20 ppm were obtained for the elements of environmental concern. The effect of increasing lead concentration on arsenic detectability was quantified. Using the EDXRF method described here, reliable As results were found for those samples containing As/Pb concentration ratios above 0.083. Accuracy and precision for the analytes of interest using the EDXRF method was shown to be comparable to results obtained by independent analysis. Comparable results for Cd, As, Pb, and Zn between independent and EDXRF methods validates the use of EDXRF analysis for hazardous waste site investigation and remediation.

ACKNOWLEDGMENT

The author would like to acknowledge James P. Walsh and Associates for site sampling and providing the independent analysis data.

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- (1) Vincent, H.; Field Screening Methods for Hazardous Waste Site Investigations Symposium Proceedings, (1988), 61.
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- (5) Criss, J.W.; Birks, L.S.; "Calculation Methods for Fluorescent X-ray Spectrometry-Empirical Coefficients vs. Fundamental Parameters", Analytical Chemistry, 40, (1968), 1080.
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Last modified: 11/02/98

Vasquez Boulevard	& I-70 - Phase III
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DRAFT- Do Not Cite

Appendix F:

Data Management Plan Additions

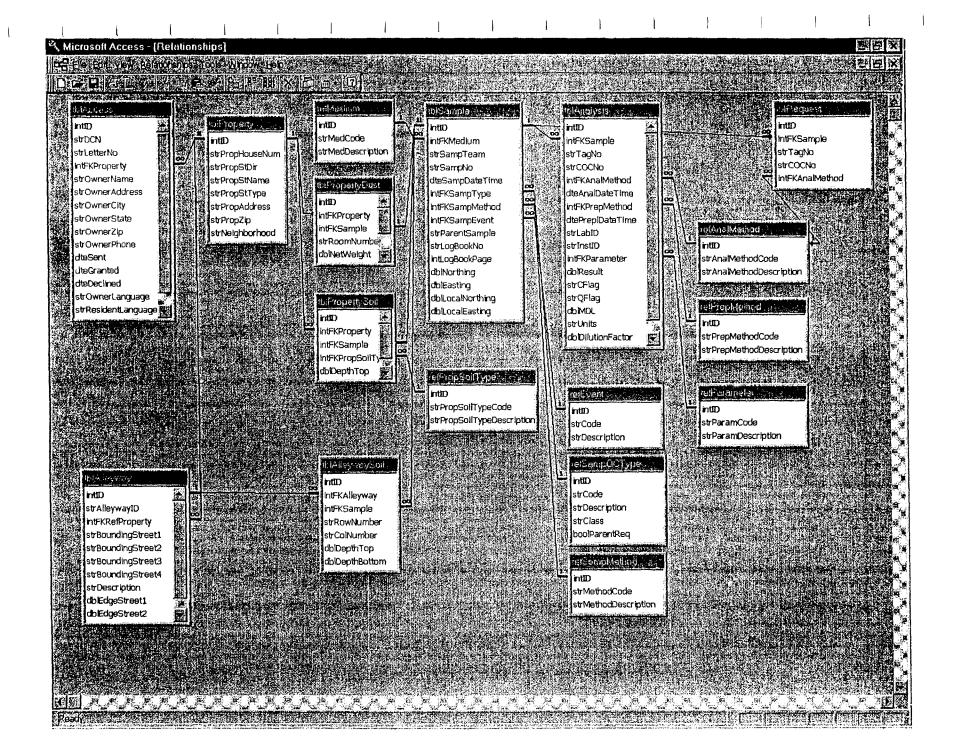


Table:

tblProperty

Description: **Property Information Table**

Order	Field Name	Caption	Field Description	Format	Valid Values
1	intPropID	AutoNumber	Unique Property ID	LongInteger	
2	strPropHouseNum	House Number	House number for subject property	C5	
3	strPropStDir	Street Direction	Street direction for subject property	C1	"S","W"," "
4	strPropName	Street Name	Street name for subject property	C35	
5	strPropStType	Street Type	Street type for subject property	C5	
6	strPropZip	Zip Code	Zip code for subject property	C5	
7	strNeighborhood	Neighborhood	Neighborhood	C20	Neighborhoods

Table: Description:

tbiSample Master Sample Log

Descrip	uon.	Master Sample Log			
Order	Field Name	Caption	Field Description	Format	Valid Values
1	intID	AutoNumber	System Generated Unique Record Identifier	AutoNumber	
2	intFKMedium	Media Code	Foreign Key to Media Code	LongInteger	refMedium
3	strSampTeam		Sampling Team	C10	
4	strSampNo			C15	Specified in Field Sampling Plan
5	dteSampDateTime	Sample Date	Date sampled in the field	Date/Time	MM/DD/YY 24:00
6	intFKS2mpType	Sample Type	Foreign Key to Sample Types	C2	refSampType
7	intFKSampMethod	Sample Method Code	Foreign Key to Sample Methods	C2	refSampMethod
8	intFKSampEvent		Sampling Event	C20	"PHASE III"
9	strParentSample	Parent Sample Number	Parent Sample Number	C15	
10	strLogBookNo	Log Book	Log Book Number	C10	
11	dblNorthing		For each sample, using the following GPS coordinate parameters: UTM, Zon	Double .	
l			13, NAD 27. Note - Units = meters.		\
12	dblEasting	Easting	For each sample, using the following GPS coordinate parameters: UTM, Zon	Double	
l			13, NAD 27. Note - Units = meters.		
13	dbll.ocalNorthing	Local Northing	Local Northing Coordinate (ft.)	Double	
14	dblLocalEasting	Local Easting	Local Easting Coordinate (ft.)	Double	

Table:

tbiAccess

Description: Access Agreement Letter Tracking

Order	Field Name		Field Description	Format	Valid Values
1	intID	AutoNumber	System Generated Unique Record Identifier	AutoNumber	
2	strDCN	DCN	Document Control Number	C10	
3	strLetterNo	Letter Number	Letter Number	C10	
4	intFKProperty	Property Address	Foreign Key to Property Address	LongInteger	tblProperty
5	strOwnerName	Owner Name	Owner Name	C50	
6	strOwnerAddress	Owner Address	Owner Address	C50	
7	strOwnerState	Owner State	Owner State	C50	
8	strOwnerPhone	Owner Phone	Owner Phone	C2	
9	dteSent	Date Sent	Date Access Agreement Letter Sent	Date/Time	
10	dteGranted	Date Granted	Date Access Granted	Date/Time	
11	dtcDeclined	Date Declined	Date Access Declined	Date/Time	
		Owner Language	Owner Language Preference	C10	English, Spanish, Other
13	strResidentLanguage	Resident Language	Resident Language Preference	C10	English, Spanish, Other
14	strComments	Comments	Explanation or comments regarding authorization of sampling	C200	

Table:

tblRequest

Description: Requested Laboratory Analysis - Used to generate Chain-of-Custody Report

			X		
Order	Field Name	Caption	Field Description	Format	Valid Values
1	intlD	AutoNumber	System Generated Unique Record Number	AutoNumber	
2	intFKSample	Sample Number	Foreign Key to Sample Table	LongInteger	tblSample
3	strTagNo	Tag Number	Tag Number	C15	
4	strCOCNo	COC Number	COC Number	C15	
5	intFK Anal Method	Method Requested	Foreign Key to Analytical Method Table	LongInteger	refAnalMethod

Table:

tblPropertySoil

Description: Property Surface Soil Sample Attributes

Deacription.		1 Toperty Surface Son Sample At	indatea			
Order	Field Name	Caption	Field Description	Format	Valid Values	
	1 intID	AutoNumber	System Generated Unique Record Identifier	AutoNumber		
	2 intFKProperty	Property Address	Foreign Key to Property Table	Long Integer	tblProperty	
	3 intFKSample	Sample Number	Foreign Key to Sample Table	Long Integer	tblSample	
	4 infFKPropSoilType	Property Soil Type	Location Type (Yard, Garden, Park, etc.)	Long Integer	refProperySoil	
	5 dblDepthTop	Top of Soil Depth	Top of Soil Depth (in)	Double	0"	
\	6 dblDepthBottom	Bottom of Soil Depth	Bottom of Soil Depth (in)	Double	2"	

Table:	refAnalN	Method

Col	um	าทร

Name Type	Size
intID Number (Long)	4
strAnalMethodCode Text	10
strAnalMethodDescription Text	50

Table: refEvent

Columns

Name	rype	Size
intlD	Number (Long)	4
strCode	Text	10
strDescription	Text	50

Table: refMedium

Columns

Name	Туре	Size
intID	Number (Long)	4
strMedCode	Text	2
strMedDescription	Text	50

Table: refParameter

Name		Туре	Size
intID	•	Number (Long)	4
strParamCode		Text	10
strParamDescription		Text	50

Table: refPrepMethod				
Columns				
Name		Туре	Size	
intID		Number (Long)	0.20	4
strPrepMethodCo		Text		10
strPrepMethodDe	escription	Text		50
Table: refPropSoilType				
Columns				
Name		Туре	Size	
intID strPropSoilTypeC	'ode	Number (Long) Text		4 2
strPropSoilTypeD		Text		50
T. 1.1 10 . 14 . 1 1				
Table: refSampMethod				
Columns				
Name		Туре	Size	
intID		Number (Long)		4
strMethodCode	Al a m	Text	٠	3
strMethodDescrip	tion	Text		35
Table: refSampQCType				
Columns				
Name		Туре	Size	
intID strCode		Number (Long) Text		4 255
strCode strDescription		Text		∠55 255
strClass		Text		255
boolParentReq		Yes/No		1

Table: tblAccess

Columns

Туре	Size
Number (Long)	4
Text	10
Text	10
Number (Long)	4
Text	50
Text	50
Text	50
Text	2
Text	10
Text	12
Date/Time	8
Date/Time	8
Date/Time	8
Text	50
Text	50
Text	200
	Number (Long) Text Text Number (Long) Text Text Text Text Text Text Text Text

Table: tblAlleyway

Туре	Size
Number (Long)	4
Text	10
Number (Long)	4
Text	50
Number (Double)	8
Number (Double)	8
Number (Double)	8
	Number (Long) Text Number (Long) Text Text Text Text Text Text Text Number (Double) Number (Double)

Tahl	۰ م	thiAi	levw	avSoil

Column	s

Name	Type	Size
intID	Number (Long)	4
intFKAlleyway	Number (Long)	4
intFKSample	Number (Long)	4
strRowNumber	Text	50
strColNumber	Text	50
dblDepthTop	Number (Double)	8
dblDepthBottom	Number (Double)	8

Table: tblAnalysis_

Columns

Name	Туре	Size
intID	Number (Long)	4
intFKSample	Number (Long)	4
strTagNo	Text	15
strCOCNo	Text	15
intFKAnalMethod	Number (Long)	4
dteAnalDateTime	Date/Time	8
intFKPrepMethod	Number (Long)	4
dtePreplDateTime	Date/Time	8
strLabiD	Text	15
strinstID	Text	15
intFKParameter	Number (Long)	4
dbiResult	Number (Double)	8
strCFlag	Text	1
strQFlag	Text	3
dblMDL	Number (Double)	8
strUnits	Text	10
dblDilutionFactor	Number (Double)	8
strBatchNo	Text	15

Table: tbiProperty

Name	Туре	Size
intID	Number (Long)	4
strPropHouseNum	Text	5
strPropStDir	Text	1
strPropStName	Text	3 5
strPropStType	Text	5
strPropAddress	Text	50
strPropZip	Text	5
strNeighborhcod	Text	30

Table: tbiPropertyDust

Col	umns
-----	------

Name	Туре	Size
intID	Number (Long)	4
intFKProperty	Number (Long)	4
intFKSample	Number (Long)	4
strRoomNumber	Text	5
dblNetWeight	Number (Double)	8
dbiTotalArea	Number (Double)	8
dblRoomTemp	Number (Double)	8
db!RoomHumidity	Number (Double)	8

Table: tblPropertySoil

Columns

Type	Size
Number (Long)	4
Number (Double)	8
Number (Double)	8
	Number (Long) Number (Long) Number (Long) Number (Double)

Table: tblRequest

Name	Type	Size
intID	Number (Long)	4
intFKSample	Number (Long)	4
strTagNo	Text	15
strCOCNo	Text	15
intFKAnalMethod	Number (Long)	4

Name	Туре	Size
intID	Number (Long)	4
intFKMedium	Number (Long)	4
strSampTeam	Text	10
strSampNo	Text	15
dteSampDateTime	Date/Time	8
intFKSampType	Number (Long)	4
intFKSampMethod	Number (Long)	4
intFKSampEvent	Number (Long)	4
strParentSample	Text	15
strLogBookNo	Text	8
intLogBookPage	Number (Long)	4
dblNorthing	Number (Double)	8
dblEasting	Number (Double)	8
dblLocalNorthing	Number (Double)	8
dblLocalEasting	Number (Double)	8

Attachment 1:

Screening Level Evaluation of Risks from Acute and Subchronic Exposure to Arsenic in Soil

ATTACHMENT 1

SCREENING LEVEL EVALUATION OF RISKS FROM ACUTE AND SUBCHRONIC EXPOSURE TO ARSENIC IN SOIL

Basic Equations

In most cases, risk to humans due to arsenic contamination of soil are driven by the estimated risk of cancer following long-term (e.g., 30-year) exposure. Based on standard USEPA default exposure assumptions and a target excess cancer risk level of 1E-04, the screening level Risk-Based Concentration for arsenic in soil is 43 ppm (EPA Region III table). It is important to note that this is a default value that does not include any site-specific information, and that the final chronic RBC for soil at the VBI70 site will be developed only after the risk assessment is complete, and that all reliable site-specific information will be included.

In some cases (where levels of arsenic in soil are especially high), risks of noncancer effects from subchronic and/or acute exposures to arsenic in soil might also be of concern. The basic equations for assessing non-cancer and cancer risks from acute, subchronic, and chronic exposure are shown below:

$$HQ(a) = C(a) * HIF(a) / RfD(a)$$

$$HQ(sc) = C(sc) * HIF(sc) / RfD(sc)$$

where:

HQ = Hazard Quotient, either for acute (a) or subchronic (sc) exposure

C = Exposure point concentration, either for acute (a) or subchronic (sc) exposure

HIF = Human Intake factor, either for acute (a) or subchronic (sc) exposure

RfD = Reference Dose, either for acute (a) or subchronic (sc) exposure

Residential Exposure Parameters

There are no standard default exposure parameters for calculating subchronic and acute exposures of residents to soil, so the following factors were selected based on professional judgement. All are based on an assumed exposure of a residential child to soil in the yard of the home.

Parameter	Units	Acute	Sub-Chronic
Soil Intake	mg/event	2,000	500
Body weight	kg	15	15
Exposure frequency	days	1	30
Exposure duration	days	1	120
HIF	kg/kg-d	1.3E-04	8.3E-06

Toxicity factors

Acute and sub-chronic oral toxicity factors for arsenic are summarized below:

Duration	RfD (mg/kg-d)	Source
Acute	1E+00	ATSDR 1993
Sub-chronic	6E-03	USEPA 1995

Screening Level RBCs for Acute and Sub-Acute Exposures

Based on these inputs, screening-level RBCs for acute and subchronic exposure are:

RBC(acute) = 7,500 mg/kg RBC(subchronic) = 720 mg/kg

It is important to stress that these screening level RBCs do not incorporate any site-specific information, and that these values may not be equal to the final RBCs developed for the site after all reliable site-specific data have been incorporated.

Application of Acute and Sub-chronic RBCs

An important attribute of the RBCs for subchronic and acute exposure is that they apply to exposure areas that are smaller than for chronic exposure. For example, subchronic exposure might occur in a preferential play area, perhaps 1/5 of the size of the yard, while acute exposure might occur at any location within the yard.

If a yard were sampled by collection of multiple grab samples of soil, the approach for applying the subchronic and acute RBCs would be as follows:

Duration	Test	Interpretation
Acute	Compare acute RBC to maximum value	Max <= RBC = acceptable
		Max > RBC = potentially unacceptable
Subchronic	Divide yard into fifths, identify that which is most contaminated.	Highest mean <= RBC = acceptable
	Compare sub-chronic RBC to mean concentration	Highest mean > RBC = potentially unacceptable

At the VBI70 site, a total of 30 grab samples will be collected from each yard. However, these will not be analyzed individually, but will be composited into 3 sets of 10. Thus, direct application of the evaluation procedure above is not possible. However, it is possible to determine whether a yard that passed the test for chronic risk could have contamination levels that failed either the acute or the subchronic test, as follows.

Acute Evaluation

Consider a yard in which 29 of the 30 total samples collected at a property were at background levels (about 20 ppm), and one sample was at the level of acute health concern (7,500 ppm). Then, the expected values for each of the three 10-point composites and the grand mean across the 3 composites would be:

Composite 1: (9*20 ppm + 1*7500 ppm) / 10 = 768 ppm

Composite 2: (10*20 ppm) / 10 = 20 ppmComposite 3: (10*20 ppm) / 10 = 20 ppm

Grand mean = (768 ppm + 20 ppm) / 3 = 269 ppm

Thus, the lowest grand mean that could be achieved if there were even one location in the yard which exceeded the acute RBC is 269 ppm. This value is substantially higher than the default chronic RBC (43 ppm), so any property which passes the test for chronic risk will also pass the test for acute risk.

Sub-chronic Evaluation

Consider a yard in which 1/5 of the area was at a level of sub-chronic concern (720 ppm), while the remainder was un-impacted (20 ppm). Then, the expected values for each of the three 10-point composites and the grand mean across the 3 composites would be:

Composites (all): (8*20 ppm + 2*720 ppm) / 10 = 160 ppm

Thus, the lowest value that the composites (either alone or combined) could have if one fifth of the yard exceeded the sub-acute RBC is 160 ppm. This value is substantially higher than the default chronic RBC (43 ppm), so any property which passes the test for chronic risk will also pass the test for sub-chronic risk.

Conclusion

Available data support the view that properties which have been contaminated with arsenic are not uniformly contaminated, and that some sub-locations within a yard may be substantially more contaminated than the yard-wide average. In such cases, preferential exposure at these "hot-spots" could conceivably be of acute or sub-chronic health concern. However, any property that has at least one sample location that exceeds the acute RBC for arsenic, or that has a sub-area (1/5th the size of the yard) that exceeds the sub-chronic RBC for arsenic, will automatically fail the test for chronic concern. Thus, application of a single test (comparing the yard-wide average to the chronic RBC) will identify all properties that are of concern for acute, sub-chronic and/or chronic health concern. Use of the 95th upper-confidence limit of the mean as the test statistic provides an even wider margin of safety for all three tests.

References

ATSDR. 1993. Toxicological Profile for Arsenic. April 1993. USEPA. 1995. Subchronic Reference Dose for Arsenic. Prepared by Robert Benson, Ph.D. September 12, 1995.